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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–7 of the January 1958 and pages 285–290 of the July 1957 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

Some Aspects of Bleaching with Hydrogen Peroxide and with Peracetic Acid — *L. Chesner and G. C. Woodford*

The Dyeing of Fast-to-washing Colours on Wool Yarn — *J. F. Gaunt*

The Dyeing of Acrilan Acrylic Fibre — *H. R. Hadfield and M. Sokol*

The Work of a Colourist in a Dyehouse Laboratory — *J. T. Lynes*

COMMUNICATIONS

The Dyeing of Secondary Cellulose Acetate with Disperse Dyes
VII— A Comparison with their Solubility in Organic Solvents — *C. L. Bird*

The Latest Trends in the Development of the Field of Azoic Dyes — *M. Hückel*

EXPLANATORY PAPER ON MODERN THEORY

Surface Activity, Solution, and Adsorption — *C. H. Giles*

THE JOURNAL
OF THE
Society of Dyers and Colourists

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Proceedings of the Society

The Manufacture of Coated Fabrics
The Processes used and their Bearing on the Choice of Pigments

H. Shepherd

Meeting of the West Riding Section held at the Hotel Metropole, Leeds, on 22nd November 1956, Mr. C. W. Green in the chair

Methods and processes used in the coating of fabrics with linseed oil, india-rubber, nitrocellulose, and vinyl resin compositions are described. The preparation of the base cloths and the compounding of the coating media are described, together with spreading and calendering techniques of application. Embossing and decorative finishing are outlined, and recommendations on the choice of colouring matters are given.

Since man first began to use woven fabrics certain inherent disadvantages in their properties have been recognised. Although their lightness in weight and flexibility made them preferable to skins, tanned or untanned, as clothing and tent coverings, their inherent porosity, whilst desirable in fine weather, was a source of discomfort in the rain. Very early in history, therefore, attempts were made to overcome the water or air permeability of woven fabrics by coating them with various substances.

Modern development of coated fabrics probably dated from the application of linseed oil compositions to fine cloths in the manufacture of oilskins for protection against the wet. A further advance led to the production of linseed-oil-coated fabrics as a substitute for leather upholstery, and to linoleum as a floor covering. The use by Mackintosh of india-rubber as a coating for fabrics introduced techniques which were later applied to nitrocellulose (cellulose nitrate) and other plastics. It was only with the use of coated fabrics as furnishing materials that the question of the colour of the coating assumed importance, for, although waterproof clothing was produced in many shades, these were mainly achieved by dyeing the base fabric, the coating being either sandwiched between two cloths or worn on the inside of the garment.

For normal peacetime purposes the colour of coated fabrics is almost invariably decorative in function, and the method of achieving the desired effects depends mainly on the type of coating and its intended end-use.

I Processes

In the present paper it will be assumed that the processes normally encountered in the dyeing and finishing of textiles are well known to the reader and that no detailed description of them is necessary. Those methods and techniques which are peculiar to the coating industries require somewhat fuller treatment: the coating of fabrics entails processes for— (1) the preparation of the fabric to be coated, (2) the preparation of the coating medium, (3) the application of the coating to the fabric, and (4) the decoration of the fabric thus coated.

1. Preparation of the Fabric

The processes employed in (1), as stated above, are those normally used in the textile industry for cropping, firing, scouring, dyeing, and stretching.

2. Preparation of Coating Media

For the preparation of the dopes the machinery and techniques employed vary, naturally, with the type of medium to be used, but in general the following equipment is necessary—

For the dispersion of pigments in plasticisers a triple roll mill (Fig. 1) and a colloid mill of the rotating-cone type are favoured.

In the triple roll mill a rough mixture of powdered pigment and plasticiser is fed into the nip between the slow back roll and the medium-speed middle roll, which also has a lateral motion to increase the shearing action. It then passes forward to the nip between the middle roll and the high-speed front roll, where the shearing action is continued, the dispersed product being

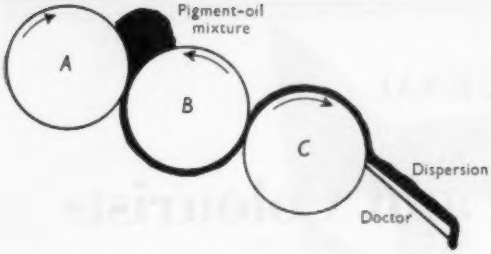

FIG. 1—Triple Roll Mill

collected from the front roll by a closely fitting doctor blade leading to a trough, down which it slides to the storage bin.

The colloid mill consists of a cone spinning at high speed close to a coaxial ring of carborundum, so shaped that the pigment–plasticiser mixture passes downwards through a decreasing gap between the rotor and the stator, and is discharged from the periphery of the cone into a collecting ring trough, whence it flows through a spout into a collecting bin.

The dispersion made by either of these two methods is tested for fineness on a Hegmann gauge, which consists of a finely machined wedge-shaped trough along which a sample of the pigment dispersion is drawn by means of a machined edge scraper, until scratches in the dispersion are observed. The trough varies in depth between 0·004 in. at one end and nil at the other, and is marked usually at thousandth and half-thousandth inch stages of depth along the length of the trough. The position at which the scratches start indicates the size of the largest particle of the sample, which for practical purposes should be no more than 0·001 in. If the dispersion is not satisfactory, further passes through the mill are given until the required fineness is achieved.

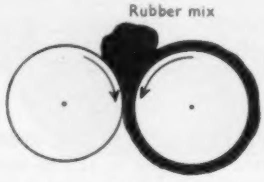

FIG. 2—Twin Roll Mill

Fig. 2 illustrates the twin roll type of mill, which is used in the compounding of rubber doughs. It consists of two hollow steel rolls, revolving in opposite directions, which can be adjusted so that whatever is placed in the nip is drawn down between them. A tearing and squeezing action is exerted, which develops heat in the mass and breaks down its original structure. The development of heat can be assisted or controlled by provision of steam heating and water cooling services through the roll bearings.

Vertical mixers of the type shown in Fig. 3 are used in the preparation of basic nitrocellulose masses or dopes. The arms revolve quite slowly, working the nitrocellulose into intimate contact with the solvents.

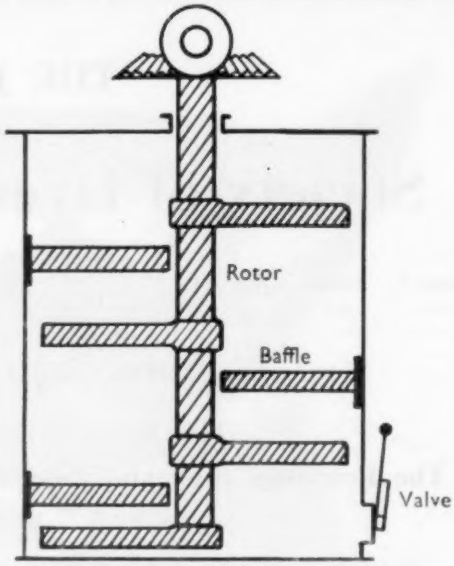

FIG. 3—Vertical Mixer

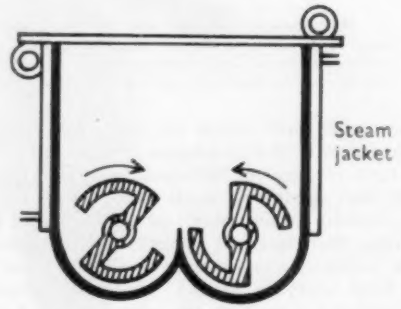

FIG. 4—Z-Blade Mixer

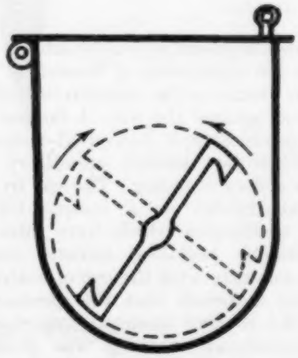

FIG. 5—Overarm Mixer

For the preparation of coloured nitrocellulose dopes it is usual to take the unpigmented basic dope and colour it by incorporating the pre-dispersed pigment in mixers of the Z-blade or overarm type (Fig. 4 and 5). When provided with heating jackets these mixers may also be used in the preparation of linseed-oil masses and varnishes.

The preparation of polyvinyl chloride spreading paste is also carried out in the Z-blade or overarm-type mixer.

The action of these types of mixers is a combination of shearing and stirring. The blades revolve so that there is little clearance between them and the walls, whilst the contra-rotation of the shaped blades moves the contents from side to side and from end to end of the mixer.

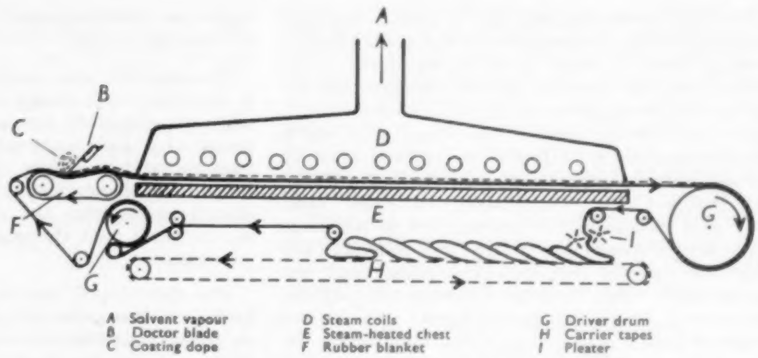

FIG. 6— Spreading Machine— Continuous Coating

For all types of finishing lacquers, whether for use with rubber, linseed oil, nitrocellulose, or vinyl coatings, the dispersion of the pigment in the lacquer is usually carried out by means of ball mills or efficient high-speed stirring machines.

In the preparation of polyvinyl chloride mixes for application by calendering a ribbon type of mixer and a Banbury mill are used, the former to incorporate the resin, plasticiser, and pigment into a uniform dry dispersion, the latter to flux this dry dispersion into a rubberlike plastic mass for feeding into the calender.

The ribbon mixer takes the form of a U-section trough closed at both ends in which a helical metal ribbon, supported on a horizontal shaft, revolves in such a way that the contents are carried up the walls of the trough and then allowed to fall, at the same time moving forward slowly. By this means the dry powders are thoroughly blended, and liquid plasticisers can be incorporated, provided that, as is the case with calender mixes, the proportion of plasticiser is insufficient to wet out the powders completely, and the resin is of a type which does not "paste up" at ordinary temperatures. A feature of such a mixer is that little or no shearing action takes place in it, and consequently the heat developed is negligible.

The Banbury-type mixer submits its contents to a severe kneading action by means of powerful Z-section rotors revolving in a steam-jacketed chamber. The mass is forced down between the rotors by a ram operated by compressed air, and is discharged through a door in the bottom of the chamber directly on to a large twin roll mill heated by steam, where it is kept hot until sheeted off for immediate transfer to the calender.

3. APPLICATION TO FABRIC

The application of coating to fabric is carried out by either spreading or calendering. Various types of spreading machines are used, depending upon the coating to be applied. They all consist essentially of a coating knife, under which the substrate is drawn and which smears on a thin film of coating. The coated substrate is then passed through a device for removing solvents or otherwise hardening the applied film to render it sufficiently strong to receive, if necessary, further coats.

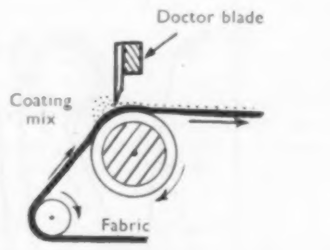

FIG. 7A— Roller Head

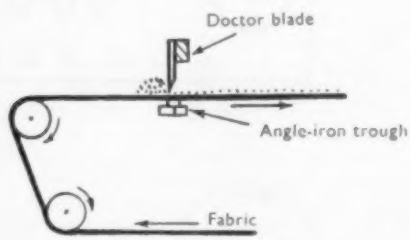

FIG. 7B— Angle-iron Head

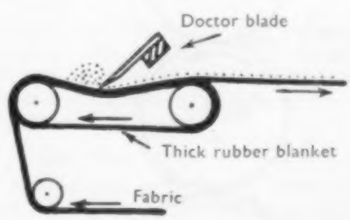

FIG. 7C— Blanket Head

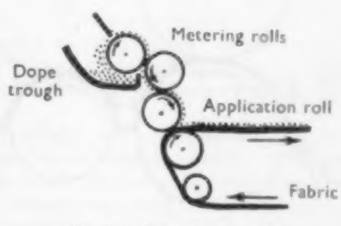

FIG. 7D— Reverse-roll Coater

A typical spreading machine is shown diagrammatically in Fig. 6, arranged for the application of several coats to fabric sewn in the form of an endless belt; the machine may be arranged also for the continuous application of a single coat. Fig. 7 shows the different types of spreading heads, which may be classified according to the method whereby the substrate is supported against the doctor blade. For instance, Fig. 7A illustrates a roller head such as is used in rubber coating, the doctor blade being superimposed on a hard rubber roller and the substrate passing between the roller and the doctor blade. Fig. 7B shows a system whereby the substrate is supported merely by its own tension whilst it passes over a trough and underneath the doctor blade. This is commonly known as an *angle-iron spreading head*, from the material used to form the trough.

Probably the most useful type of spreading head is, however, that shown in Fig. 7C, in which the cloth is supported against the doctor blade by means of a thick continuous rubber blanket which runs round two rollers, one in front of and one behind the doctor knife. This rubber blanket is driven and carries the cloth with it under the doctor blade with the minimum of tension on the substrate.

For thin dopes, use is often made of the air knife, which consists of a nozzle or series of nozzles so placed that they remove the surplus of a layer of dope, which has been previously applied to the substrate running beneath it. By this means very thin coatings may be applied.

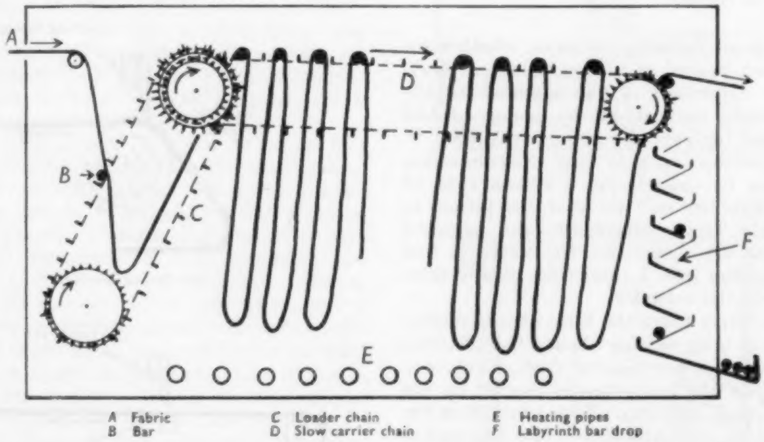

FIG. 8— Festoon Stove

There is also the reverse-roller coating system, in which a previously metered layer of the dope is applied to the fabric by means of a roller running in the reverse direction to that of the substrate (Fig. 7D).

The removal of solvent, or setting of the film, may be carried out in a tunnel heated by steam pipes or plates above or below the coated fabric. It is, however, necessary in certain instances to sling the coated fabric in loops in a large stove of the type shown in Fig. 8.

Calendering is carried out on a machine of the type shown in Fig. 9. This shows a four-bowl superimposed calender into which the prepared coating mixture is fed between bowls 1 and 2, where it is squeezed out into the form of sheet, which, after passage through

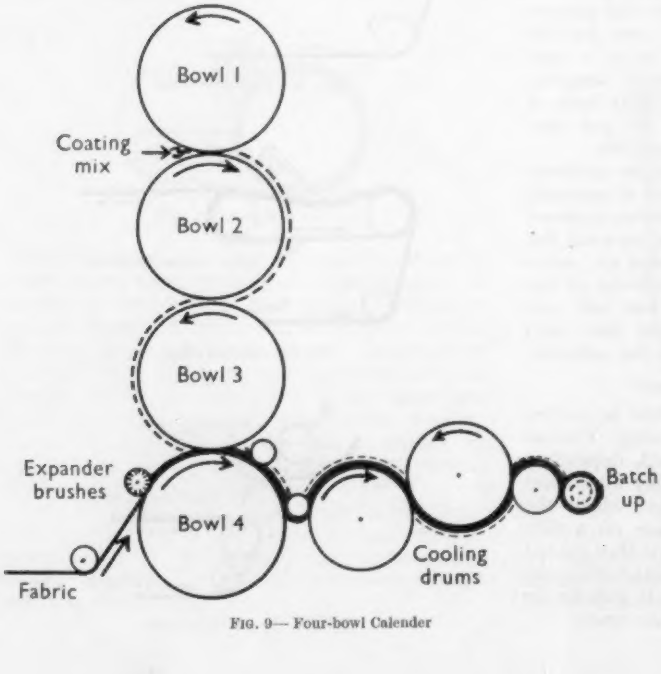

FIG. 9— Four-bowl Calender

the nip between bowls 2 and 3, meets the fabric and is pressed into intimate contact with it in the nip between bowls 3 and 4. Depending on the type of coating and the particular manufacturing process, the calender may have three or four bowls, which may be arranged either vertically, in the form of a Z, or as an inverted L. They may also be so arranged that a rubbing action takes place where the coating sheet meets the cloth, and this frictioning gives a better finish and more intimate contact of the coating with the cloth.

4. DECORATION

The coated fabric may be decorated in many ways, and combinations of the various processes are often employed. Grains are applied to the surface by means of embossing machines. These may be of the roller type (Fig. 10) or the plate-press type (Fig. 11). If it is necessary to heat the coating before application of the grain, this is usually done by passing it underneath an infrared heater, fixed immediately prior to the embossing nip, and in order to assist in the setting of the grain it may be necessary to have a cooling drum just after this nip.

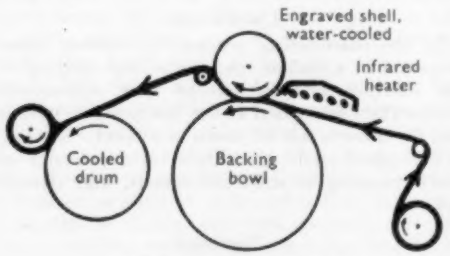

FIG. 10—Roller Embosser

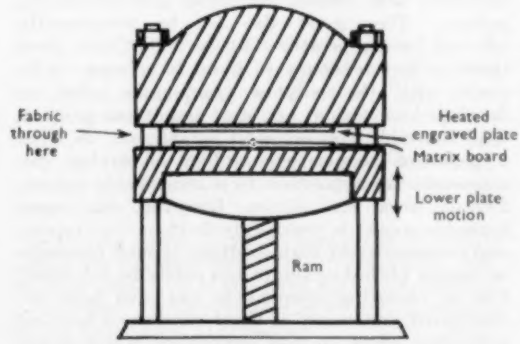

FIG. 11—Plate Embossing Press

The plate-press type of embosser is usually employed only where the grain to be applied is a true replica of a whole skin, in which the marking along one edge differs from that along the other, and this could not be conveniently engraved on a roller. Such a design, when embossed on a long length of coated fabric, shows marks where the impression of the two different edges of the plate meet. Because of the slight variations which take place in the yielding of the fabric to the tensions required, it is usual for the draw-off from the press to be so adjusted that a slight overlap takes place, and such overlap is preferable to an unembossed line running across the piece. By very careful adjustment and long experience it is possible to reduce this overlap to a minimum, and it is the object of the good embosser to keep the overlap as small as possible and yet to avoid any "miss marks".

Finishing coats of contrasting colours may be applied by spreading or printing, whilst the gloss of the finished product may be controlled by the application of dulling or brightening lacquers.

II Methods of Manufacture

1. LINSEED-OIL TYPES

Linseed-oil-coated fabrics may be of the flexible or semi-rigid types. The basis of the coating in each of these cases is a drying oil or a mixture of drying and semi-drying oils in various states of polymerisation. The film-forming medium thus resembles closely that of linseed-oil-type paints, and the pigments used for colouring are essentially the same as those employed for paint manufacture. Due regard has to be paid to the resistance of the pigments to the conditions which they have to withstand during processing.

Flexible Linseed-oil Fabrics

In the manufacture of the flexible types of linseed-oil fabrics the base cloth is usually scoured or bleached cotton cloth of the plain sheeting type.

The coating is prepared from blown, tanked, or otherwise semi-oxidised oils combined with resins into which fillers and pigments are dispersed. The mass or "varnish" is thermoplastic, and is maintained at such a temperature that it is of the consistency of treacle. It is then spread on the cloth by means of a blanket-type machine (Fig. 7c). The coated cloth passes into a hot festoon stove (Fig. 8), where the film is set by heat, assisted in certain processes by ultraviolet irradiation. Further coats are applied as required to build up the desired thickness, and a grain may be applied by embossing.

Very attractive effects are obtained by printing and coating with pigments dispersed in thin varnish, and the colours may be protected by a final coating of unpigmented clear lacquer, usually of the nitrocellulose type.

Semi-rigid (Linoleum) Fabrics

The base fabric used in the manufacture of linoleum is hessian, usually rotproofed. The coating medium is linseed oil, blown or tanked, and cooked with resins in which high loadings of cork dust, whiting, china clay, and pigments are dispersed. This thermoplastic mass is applied to the hessian by calendering, which presses the preformed film of the desired thickness into contact with the hessian as it passes through the calender nip.

Jaspé effects are obtained by first of all preforming films of desired colours. The colour films are then made into crumbs, and suitable amounts of the crumbs from each of the desired colours are mixed together whilst cold. The mixture of

crumbs is friction-calendered on to the hessian, and in this process the crumbs are squeezed out into streaks of random size and distribution, the actual effect being controlled by the art and the skill of the calender operator.

The production of inlaid patterned linoleum is achieved by sheeting out the coloured mass to the desired thickness and cutting the sheet, by means of dies, into the shapes required in each colour. These shapes are then assembled, jigsaw fashion, on the hessian as it passes over a table behind a spreading head, which applies a tacky finish to its surface to hold the shapes in place. The assembled patterned linoleum is then passed through a press or calender, which fixes the shapes firmly to the hessian and to each other. Whether patterned or otherwise, the linoleum is usually given a final protective coat of clear varnish and is polymerised or dried in a festoon stove.

Pigments

The pigments used for colouring the base coatings must be compatible with the medium used. They should not produce undue "livering", and should be resistant to light. They must not be permanently affected by heat, and should be resistant to the action of the usual washing techniques, in which mild alkalis and detergents are used. Where possible, inorganic pigments are favoured, such as the iron oxides (C.I. Pigment Yellow 42), lithopone (C.I. Pigment White 5), chrome yellows (C.I. Pigment Yellow 34), and ultramarines (C.I. Pigment Blue 29), but for special shades, and in the finishing varnishes, organic lakes of good light fastness may be used, provided that they contain no water-soluble dyes.

2. RUBBER PROOFINGS

Rubber coatings are applied to fabrics either by spreading latex or solutions or by calendering a thermoplastic mix.

Latex Spreading

Rubber latex is an aqueous dispersion of rubber, containing either 30% or 60–70% of rubber. For ease of spreading it has usually to be thickened by admixture with an agent such as casein or one of the cellulose ethers or esters, and at the same time any colouring matters, fillers, and vulcanising agents are also incorporated in the form of dispersions in water. A churn mixer is usually employed for this process. Spreading is effected by means of the conventional rubber roller machine, and drying and vulcanising take place as for solution-spread fabric.

Solution Spreading

In the preparation of rubber solutions it is usual first of all to break down, on a twin roll mill (Fig. 2), the rubber as received, until, by the application of friction and pressure, the rubber becomes plastic and can be led round one of the rolls to form a continuous band. This process is assisted by the addition of softeners such as pine oil and hydrocarbon oil, which are added when the rubber is on the mill. Pigments, fillers, antioxidants, and sulphur are milled into the plastic rubber, and mixing is facilitated by cutting the sheet from one side of the roll and passing it over on to the other at frequent intervals. Vulcanisation accelerators are not added at this stage to mixes which contain sulphur. When the mixing is completed, the rubber sheet is removed from the mill and put to soak in solvent. After sufficient swelling time has been allowed, the solution is completed in a pug mill or mixer, or on a separate twin roll mill, by which the rubber mix is thoroughly dispersed in the solvent. Finally the vulcanisation accelerators are mixed in thoroughly, usually as a solution of a "master batch", and the rubber dough is ready for spreading. The spreading is carried out on a roller-head machine (Fig. 7A), and the water from the latex or the solvent from the solution is evaporated from the film by passing over a steam-heated chest. The spread fabrics may then be cured at 130–140°C. for 1–1¼ hr. (heat cure) or by exposing to sulphur monochloride and ammonia (cold cure), or the rubber may be so compounded that the heat used for drying the spread coating may be sufficient to initiate a spontaneous cure (room-temperature cure).

Calendering

In the calendering process the rubber mass is prepared in a similar manner to that employed in the solution process, except that vulcanisation accelerators are added at the last possible moment, and the mass is not let down in solvent. This mass is frictioned on to the fabric on a calender with bowls running at different speeds, and cured by heat.

Pigments

In selecting colorants for rubber media, it is necessary to have pigments which are resistant to ammonia and alkalis, including (for latex) 5% potash. They must also not be permanently affected by temperatures up to 200°C., nor must there be any tendency to bleed in solvents or to react with the sulphur compounds used or developed in curing. A most important point is that ionisable copper must be absent, as even 5 parts of copper per million of rubber can accelerate decomposition to a remarkable extent. Recent work has shown, however, that non-ionisable copper is practically inert in this respect, and comparatively high loadings of such pigments as copper phthalocyanines can safely be tolerated. Use of chelating compounds has also been investigated, but many of these substances have an activating effect on iron compounds which would otherwise be inert, and care must therefore be taken if it is desired to protect the rubber from ionisable copper by the use of a sequestering agent.

The coated rubber fabric can be decorated by printing and embossing, the printing inks employed being usually dispersions of pigments in latex or shellac varnish.

Base Fabrics

As previously noted, the dyeing of the fabric is of importance in rubberproofing, in that it is usually the dyed fabric which is exposed to view. For

cotton fabrics sulphur and vat dyes are favoured. For viscose rayon it is usual to have the yarn spun-dyed. The remarks about ionisable copper which were made in reference to pigments apply with equal force to the fabrics, and strict control of the copper content of the cloths used is imperative.

3. NITROCELLULOSE COATINGS

Base Fabrics

In the manufacture of nitrocellulose-coated fabrics it is necessary, first of all, to consider the preparation of the base cloth, which is usually a cotton sheeting, twill, or moleskin. As received from the weavers, these cloths contain a proportion of the size applied to facilitate weaving, and this may itself consist of starch, fatty matter, and a filler such as china clay. It is desirable to remove as much of this size as possible in order to facilitate level dyeing, but special attention must be paid to the removal of the fatty matter, especially if this is of the tallow variety.

Residual fatty matter in the cloth gives rise to bloom on the surface of the finished coated fabric. The cloth is therefore desized and thoroughly scoured before dyeing. It is also found an advantage in many cases to crop or fire the cloth to remove the knots and slubs, etc., whilst for specially fine cloths and light-weight coatings the cloth should be carefully picked. The dyes used should be insoluble in solvents for nitrocellulose, and, for normal upholstery and bookbinding grades, direct dyes are sufficiently stable. Where in the finished product it is intended that the cloth side should be visible, vat dyes specially selected for their fastness to light and their insolubility in nitrocellulose solvents should be used. After dyeing, the base cloths are mangled and dried on a stenter to maintain the necessary width.

Preparation of Coating Medium

The coating dope is prepared by dissolving nitrocellulose in the form of either virgin nitrocotton or recovered celluloid, in solvents such as acetone, methylated spirit, *iso*propyl alcohol, benzole, toluole, and butyl acetate: each manufacturer has developed his own mixture of solvents to suit his own conditions.

Softening agents or plasticisers must be used to obtain the required pliability for the coating film. Of these castor oil is the most favoured, modified as necessary by the addition of triaryl phosphates or the lower-alkyl phthalates.

The colouring of nitrocellulose dopes is usually achieved by mixing in a proportion of pigment predispersed in plasticiser. This dispersion of the pigments is carried out on a triple roll dispersion mill or on one of the high-speed colloid mills, and it is the usual practice for each pigment to be dispersed separately, the dispersion being stored until it is necessary to produce a matched colour. The colour matching is normally carried out by mixing the pigment dispersions together and comparing the resultant mixture with a standard mixture by spreading the two in contiguous strips on a glass slide. The nitrocellulose dope, suitably pigmented, is applied to the fabric by spreading.

Spreading

Spreading machines are of various types, but the most usual for nitrocellulose are the continuous-blanket, angle-iron, and air-knife types. Whichever type is used, application of the dope is followed by passage through a heated chamber, which drives off the solvent and leaves the pigmented and plasticised nitrocellulose film behind. Depending on the weight of the coating required, it may be necessary to apply anything between 3 and 30 coats, in which case it is desirable that the cloth should run in a continuous band round the machine. When the basic colour coats have been applied, a clear lacquer coat is given, which consists of a straight nitrocellulose solution with possibly the addition of a small amount of plasticiser to prevent such phenomena as wrinkling on drying.

Printing

From the spreading stage the nitrocellulose-coated fabric may proceed either to the printing process or directly to the embossing room. Printed designs are applied by means of an intaglio roller, the inks used being highly pigmented nitrocellulose solutions suitably balanced for rate of evaporation. One or more colours may be printed at once by the use of suitable machines. It is then usual to pass the printed material forward to the embossing room for the application of a grain.

Embossing

In the embossing department the coated fabric is given a grain by passing it through the nip between a heated metal shell bearing the milled grain and a mating compressed cotton or paper bowl. Roller embossing, as it is called, is to be preferred from the point of view of production, but its use is restricted to conventional grains. Where replicas of real skin grains have to be made, use is made of a press in which the coated fabric is pressed against a heated metal platen milled with the desired grain, by means of a hydraulic or toggle-operated ram, bearing a platen armed with paper or board on which the mate of the design has been previously worked up.

Antiquing

From the embossing process the coated fabric may, according to its end-use, be sent either directly to the inspection department or to receive a surface finish. The surface finish may take the form of a second contrasting colour run into the grain to emphasise its colour. This process, known as *antiquing* or *scumbling*, is carried out on a spreading machine, and the dopes used are thin, highly pigmented nitrocellulose solutions. It is then usual for the surface to receive a coat of lacquer, which imparts the requisite dullness or brightness to it, and serves to seal in the antiquing colour. Special finishes may also be applied by spraying the surface of the coated fabric to obtain shot or mottled effects, but in any case it is usual for the final coat to be of unpigmented nitrocellulose to give the sealing effect.

Pigments

In the choice of pigments for colouring nitrocellulose-coated fabrics the desirable features are that they should be consistent in colour, strength, and fineness, that they should not bleed in nitrocellulose solvents, that their fastness to light should be adequate, and that their moisture content should be low. If castor oil is the plasticiser, it is also necessary to ensure that the pigments do not accelerate the development of rancidity. Such pigments as Prussian Blue (C.I. Pigment Blue 27) and certain of the Brunswick Greens (C.I. Pigment Green 15) show this last characteristic to a marked degree, and, if they are used in the composition, the coating becomes either very brittle or very tacky after a comparatively short life, owing to decomposition of the castor oil by accelerated oxidation. The intermediate stage before brittleness or tackiness occurs is marked by the development of a very offensive rancid odour, and naturally the users are prone to object strongly if this happens too quickly.

Bleeding in solvents is of importance, as solvent-soluble dyes have the property of migrating to the surface of the coating, ready to be wiped off by clothing or to migrate on to anything with which they come into contact.

4. VINYL-COATED FABRICS

Of the coated fabrics produced nowadays a very large proportion are coated with pigmented and plasticised vinyl compounds based almost exclusively on polyvinyl chloride or its copolymers. Because of its high toughness and flexibility, the polyvinyl chloride type of coating has found favour for upholstery, fancy goods, and flexible floor coverings, whilst its relative non-inflammability has rendered it very attractive from the point of view of public service vehicles and aircraft and the panelling of corridors in public buildings and cinemas.

Base Fabrics

The base cloths are usually prepared by cropping or singeing to remove slubs, knots, loose threads, etc., scouring to remove size, and dyeing or bleaching. For the majority of the coated fabrics, it is unnecessary that the dyes used for the cloth should be particularly fast to light, and normal direct dyes are admirable for the purpose, as they are usually insoluble in organic solvents and do not bleed into or through the coatings applied. For certain purposes, however, it is necessary that a higher standard of dyeing should be obtained, e.g. where the cloth is to be used as a collapsible hood covering for motor cars. Here the use of vat dyes is called for, but again care has to be taken that they do not bleed through or into the coatings and that they are fast to rubbing and moisture. They should also not be sensitive to the action of heat.

Processing

Polyvinyl chloride coatings may be applied by spreading in the form of a paste or by calendering a preformed film on to the surface of the cloth.

Preparation of Medium

Depending on the process desired, the resins are chosen and mixed with plasticisers such as tritolyl phosphate, trixylenyl phosphate, dialkyl phthalates or dialkyl sebacates. The pigments used are dispersed in the chosen plasticisers, and are then mixed together to give the desired colour. The dispersion is usually carried out on a triple roll or colloid mill, and, because of the poor wetting properties of synthetic plasticisers, it may be necessary to introduce a small amount of a special wetting agent. Blown castor oil in small amounts has been found satisfactory. The mixture of pigments is then incorporated into the basic mixture of resins and plasticisers in an overarm or Z-blade mixer.

Spreading

Spreading is carried out on a continuous blanket, a roller head, or a reverse-roll coater, and the coated fabric is then led through a chamber or round a heated drum, where it is subjected to a temperature of 100–120°C., whereby it is solidified to such an extent that it does not crack on careful handling, and at this stage further coats may be added until the desired weight or thickness of coating has been applied. In some processes the coated fabric is heated to 150–180°C., which has the effect of completely gelling or curing the pigmented and plasticised resin mixture during each pass through the spreading machine. This enables the coating to withstand handling, and may, indeed, allow it to be run completely round the machine, so that it may be spread in a continuous band for the required thickness or weight to be obtained. If the coating has not been fused during spreading, it is subsequently passed through a chamber heated by either infrared elements or steam pipes until it attains the gelling temperature of 150–180°C.; or else it is heated, usually by infrared elements, for a short time immediately prior to its passage through the embossing machine.

Calendering

If the coating is to be applied by calendering, the resin–plasticiser–pigment mixture is milled in a closed and heated kneading machine of the Banbury type, and is there gelled into a coherent thermoplastic mass. This mass is then fed, whilst still hot, through the calendering rolls, and, being preformed into a sheet of the desired thickness by passage through the first series of rolls, is finally pressed on to the cloth, which is led through the bottom nip of the calender. The coating is then cooled over water-cooled rollers, and is ready for embossing. This is carried out on a machine where the coating is preheated to near gelling temperature and the design impressed on it by means of a water-cooled steel shell. Further cooling takes place after the embossing, and the coated fabric is passed forward for finishing.

Finishing

This finishing is carried out on spreading machines for the normal antiquing type of finishing, on printing machines for print finish, or the finish may be applied by spraying. The lacquers here

are themselves solutions of vinyl resins, suitably plasticised and pigmented.

Pigments

It will be seen that the pigments chosen have to withstand quite high temperatures, and they also have to withstand any acidic conditions developed by whatever slight decomposition of the polyvinyl chloride takes place during the mixing and gelling processes. They must, of course, be fast to light, and they must not bleed or migrate through the plastic coating. There are several other points to be noted about the choice of pigments, and particularly about the matching of colours, in vinyl-coated fabrics. As previously mentioned, polyvinyl chloride tends to decompose on prolonged exposure to high temperatures, and a certain amount of hydrochloric acid is generated during decomposition. A clear film of polyvinyl chloride when heated for several hours at temperatures even as low as 100°C. shows a tendency to discoloration. In order to counteract this, reagents of the type of white lead, lead stearate, or certain tin, barium, and cadmium compounds are used in small amounts in the mixture. These appear to act as acid acceptors and prevent the autocatalytic decomposition of the polyvinyl chloride. It is always safest, however, when matching a colour, to make the final adjustments subject to actual tests on the resins and plasticisers to be used, and actually to put the test matches through heating processes similar to those which they will be expected to withstand. Similarly, when checking a new pigment for suitability for use with polyvinyl chloride, tests on the actual mixings should be carried out, as the type of resin used may have a profound effect on the light fastness. Examples of the pigments used to colour vinyl compounds are —

WHITES— Titanium dioxide (C.I. Pigment White 6) of the anatase or rutile types.

Note— Whites containing zinc are liable to cause accelerated decomposition of polyvinyl chloride.

BLUES— Phthalocyanine types (C.I. Pigment Blue 15), either pure or as lakes.

Note— Ultramarines are affected by the acidic conditions which may be developed in processing or ageing, though they find a limited use in "blueing off" whites.

GREENS— Phthalocyanine types (C.I. Pigment Green 7), pure or as lakes. Mixtures of blues and chrome yellows or certain organic yellows may be used. Generally, phthalocyanine blues and cadmium yellows give very "muddy" colorations.

BLACKS— High-tinctorial Carbon Blacks (C.I. Pigment Black 7), and in certain cases Aniline Black (C.I. Pigment Black 1).

REDS— Cadmium selenides (C.I. Pigment Red 108) are fast to light and bleeding, but are "weak" and costly. Ranges of organic reds are now available which, when chosen with care, give very good results, but many are subject to migration, especially in paste-spread coatings. Scarlets may be obtained by means of tungstated chromes, which, however, have in many cases a tendency to darken on exposure to light.

YELLOWS— Cadmium yellows (C.I. Pigment Yellow 37) are good but weak and somewhat costly. Suitably selected chromes are generally suitable, and may be used subject to their tendency to darken slightly on exposure to light. Organic yellows are now on the market which show many good qualities, and whilst initially costly, this is offset by their high tinctorial value.

BROWNS— These are usually obtained with mixtures of yellows and blacks, chromes and Carbon Blacks giving a good combination. The orange and middle chromes are mainly used in this connection.

Although a pigment may be found to be suitable for use by itself, it does not follow that it may be used indiscriminately with other pigments in matching colours, and much care is necessary to ensure that the pigments used in any colour are compatible with each other, and that the mixture does not show any untoward idiosyncrasies in the coating composition.

In the field of fabric coating, the application of colour becomes of increasing importance as the education of the consumer progresses, and much time and ingenuity go into catering for a demand which only increases as attempts to satisfy it reveal new regions to be explored.

Lacunae on the coater's palette, such as the absence of really satisfactory crimson, violet, and yellow pigments at an economic price, are subjects of much research on the part of manufacturers and of much heartache on the part of the coating colourist. Great strides have been made recently, however, and there are hopes that in the near future at least some of the gaps will be filled. But then, of course, it is quite probable, and indeed desirable, that new demands will arise and new applications for coated fabrics be discovered which will beckon us to further adventures in the use of colour for their decoration.

* * *

The author wishes to offer his thanks to the directors of Armoride Ltd. for permission to publish this paper, and to Mrs. J. M. Slater for assistance in its preparation.

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(*MS. received 17th May 1957*)

. . . and inwardly digest*

S. BURGESS

Meetings of the Manchester Junior Branch held at the Manchester College of Science and Technology on 5th February 1957, Mr. J. W. Reidy in the chair; of the West Riding Section held at the Victoria Hotel, Bradford, on 14th March 1957, Mr. C. W. Green in the chair; and of the Midlands Section held at the King's Head Hotel, Loughborough, on 17th April 1957, Mr. C. A. Mills in the chair

The problem of keeping abreast of the increasing volume of technical literature is discussed, special reference being made to the technique of fast reading, and the quality of scientific writing.

This paper is more full of quotations than the old lady said *Hamlet* was. It is about reading. In the *Book of Common Prayer*, the Collect for the Second Sunday in Advent contains the following—

> Blessed Lord, who hast caused all holy Scriptures to be written for our learning: Grant that we may in such wise hear them, read, mark, learn, and inwardly digest them, ...

I hope that I shall not be considered irreverent in having taken my title from this text and extended its application to the matter which concerns us, that of trying to keep up with the spate of papers, journals, reports, and the like. That reading and study are essential will, I think, be agreed by all. There is, however, room for difference of opinion as to how much should be done and who is to do it. The critics of too much reading can call Holy Writ to their aid by quoting from the Book of Ecclesiastes [1]—

> And further, by these, my son, be admonished: of making many books *there is* no end; and much study *is* a weariness of the flesh.

I submit that no one, however great an enthusiast in the search for information, who has allowed one week's collection of journals and reports to accumulate will reject this judgment completely.

FAST READING

The problem is so widespread that "Fast Reading" is now systematically taught, and interest in it is deemed sufficient to warrant references in the daily papers and popular periodicals such as *John Bull* [2]. Much of the impetus comes from big business, initially in the U.S.A., whose executives have too much to read, spending as much as 25% of their valuable time upon it. Behind the art of fast reading is a great deal of erudite research work, starting about 1878, when a worker named Javal noticed [3] that he, "by watching a reader's eyes, found that they moved along the lines not with a smooth, steady movement, but in a series of jumps or 'saccades', separated by fixation pauses".

Other fundamental discoveries made before the end of the century were that familiar words are read in one as a whole, not by spelling out the letters; that such words are read as quickly as single letters, and that phrases consisting of several words can also be read in one. The "fixation pauses are the times of effective exposure and stimulation of the eyes, the eye movements in reading being simply quick jumps from one fixation to the next".[3]

A series of "16 Harvard reading films" has been used to train people to read quickly [4,5]. Other mechanical aids used are—

(1) A tachistoscope, which flashes numbers and words on a screen to train the eyes to take in more and so make fewer stops per line

(2) An accelerator, whose descending metal shade forces the reader to read faster and curbs the habit of reading back for missed words.

(3) A timer, which checks words-per-minute speed.

In addition, the student takes tests for "comprehension" of selected matter, usually by answering a series of yes-or-no questions.

As one would expect, the claims made for the success of fast reading methods vary rather widely. *Fortune* delightfully says [4]: "barring eye trouble or a weak vocabulary, an executive may double his non-fiction and treble his fiction reading speeds with certainly no loss, and possibly some gain in comprehension". ICI quote [5] increases in reading speed without loss of comprehension of 65–115%. Very fairly they also point out that, whilst, on the basis of initial and final tests only, the average increase in comprehension was 80·6%, the average improvement if sessions 2 and 3 of the course are compared with sessions 12–15 was 40·7%. It is emphasised that no loss in comprehension is involved, and this is supported by the fact that the highest individual reading speed in one of the courses, 555 words/min., was accompanied by 100% correct answers to questions.

On the basis of initial and final tests, ICI record [5] that the actual average reading speed of a group rose from just under 200 to about 355 words/min.

An American business magazine has been running a series of articles [6] by Dr. Nila Banton Smith, Director, Reading Institute, New York University, which are quite helpful because they print several aids and so enable one to "have a go" without assistance or mechanical aids. He says that the average reader reads about 250 words per min. Less means a slow or poor reader. Ordinarily good readers attain 500–600 words/min. Very superior readers attain 1,000, and very occasionally someone scores 1,600 or 1,800 words/min.

Using these articles I found that my normal rate for the ordinary technical article was easily increased from 230 to 320 words/min. without

* *The Assimilation of Technical Literature.*

any loss of comprehension, but I can read fiction at 416 words/min. Thus my personal attempt gave results more closely resembling those of ICI than the American figures.

It seems that there is now general agreement that fast reading can make a useful contribution to the problem of surfeit of words. The use of mechanical aids has, however, been severely criticised by two directors of the Bureau of Study Council of Harvard University. They contend that the problem is essentially mental; that readers have been too conditioned by academic educational methods to accept the authority of written words, to return (presumably to the examiner) what is read, and to skip nothing. Reading, they say, should be "an active aggressive acquisition of ideas and unless students analyse, criticise, anticipate, select, compare, rehearse, admire and (for good reason) sneer while reading, the process is an utter waste of time".

Another critic of machine-using methods contends that after their use has ceased reading speeds tend to drop, whilst if they are not used speeds continue to increase after the course is finished.

Just one other point about fast reading. In New York business executives pay $80–100 a head for their course of lessons. Fourteen 90-min. daytime lessons may cost 50 guineas in London at the Quick Reading Centre. Evening classes at the North-Western Polytechnic (London) may be had for one guinea.

INFORMATION

Any attempt to rationalise one's attitude to the problem of trying to keep reasonably well informed must include consultation with those whose profession it is to collect, sort, and distribute information— the librarians and information officers— who are, individually and collectively, the most helpful people I have ever met. Each and everyone I approached was most co-operative and forthcoming.

I was directed to a book entitled *The Royal Society Scientific Information Conference* [7], a most rewarding volume. In opening the Conference, Sir Robert Robinson, P.R.S., distinguished between strictly scientific information on the one hand and that required for social and humanitarian studies on the other, saying that, nevertheless—

> The sciences have deep human interest and are not devoid of spiritual value. The object of our Founders was declared to be the improvement of natural knowledge. By that they meant, and we still do mean, improvement and spread of knowledge of nature. Neither they could, nor we can, condone the scientific miser who investigates for his own satisfaction, or profit, and keeps the results to himself for selfish reasons, whether they be æsthetic or economic.

an admirable attitude in sharp contrast to that still occasionally to be found in industry and expressed by the phrase: "Those who know won't tell, and those who tell don't know".

Indeed, it is probable that we technologists do not appreciate how important to the research worker is this question of publication. It would seem that there is a fundamental urge in the individual to play the fullest possible part in the cross-fertilisation of ideas which is so powerful a force in "the improvement of natural knowledge". Faraday said that the three necessary stages of useful research were "begin it", "end it", "publish it". Dr. E. Marsden said that research is "30% idea, 30% experimental work, and 40% the actual writing up".

This stress upon publication can be contrasted with that of the captain of industry who said that he liked any new discoveries of his research workers to be published immediately in the high-brow scientific journals because this method of burying them ensured that he did not have to share them with his competitors.

In the Conference Lecture, Professor E. N. da C. Andrade [8] said that the spoken word is still important. Scientists should become proficient in using it. He recalled the story of the man who was asked if he could play the violin and said he did not know because he had never tried. Sir William Bragg in his early days was unimpressive, but by careful application attained high eminence as a lecturer. In ancient times, men travelled great distances to learn by means of the spoken word. The value of learning by reading should not, of course, be minimised, although the great historian Mommsen (writing of the time of Julius Caesar) does not seem to have been wholly in favour of it: "Reading had become a fashion, nay a mania . . . The superior officer was seen in the camp-tent with the obscene Greek romance, the statesman in the senate with the philosophic treatise, in his hands". The two great libraries of Alexandria were reputed to contain half a million books, which were finally destroyed by the Saracens. The Caliph replied to the general who wished to spare the books: "If these writings of the Greeks agree with the Book of God, they are useless and need not be preserved: if they disagree they are pernicious, and ought to be destroyed."

Having referred to the spoken word and made some distinction between the attitude of the technologist and that of the scientist to the question of publication, I would like to quote from a lecture on *The Technical Education of a Dyer* given to the London Section of our Society in 1905 [9]. The author also was quoting, from a lecture by a Mr. John Wallace on the general subject of technical education—

> "it was just possible that the choice of a Greek rather than a Latin root might have something to do with the popular misapprehension, for techne simply means art, and the peculiarity of all art was that it could not be communicated in writing alone, however well instructed the reader might be. In this respect it was clearly distinguished from *science*, which, as it represented exact knowledge, could be clearly and completely expressed in print. Craft was a term which is synonymous with technique and art, but the latter appeared to be the most appropriate term with which to express one of the two great divisions of our knowledge of physical work, for it expressed with equal appropriateness the skill of the painter, the smith, the gymnast, the hunter or the ploughman . . ."

The reading of books, including bound volumes of old journals, is a problem sufficiently distinct from that of keeping up with current material to justify its exclusion from this discussion. The reading of books is essentially a leisure occupation,

one which competes with more sociable activities such as conversation, television, radio, and golf, possibly in that order.

The Royal Society Scientific Information Conference was very thoroughly seized of the magnitude of the problem with which they were dealing. Sir Edward Appleton [10] said—

> If anyone set himself the task of merely reading—let alone trying to understand—all the journals of fundamental science published, and worked solidly at his task every day for a year, he would discover that at the end of that year he was already more than 10 years behind! If the same constant reader (I think we may well call him a constant reader) had included the technical literature as well, he would find himself about 100 years behind in his work after 12 months' effort!

The resources of the Manchester Public Libraries Technical and Commercial Information services include 1,150 commercial and technical periodicals taken regularly, of which 650 are technical journals. In addition, many others are obtained on occasion. This is quite apart from the purchase of every book on science and technology published in England other than trivial matter and medical literature. There are also, of course, trade papers which contain technical articles. Upon all these, plus 1,700 directories and many yearbooks, statistical publications, and other material, the Manchester Libraries have built a service (including immediate replies where necessary by telex and telephone) and offer—

> To answer specific queries from published sources on technical, scientific, and commercial subjects
> To compile reading lists on specific subjects
> To provide photocopies of non-copyright material at nominal charges
> To obtain from other sources publications not already available in the Central Library.

ICI take 1,000 journals, many of them foreign, especially German.

As regards the language in which papers are written, in 1934 Sir Charles Sherrington [11] made an examination of the *World List of Scientific Periodicals*: of the total of 28,193, 13,494 were in English, which suggests that English is and should be the scientific language of the time. According to Locke [12], however, the amount of technical material which is being published in languages other than English is tending to increase, and with it, of course, the necessity for the expensive process of translation, which is stated to cost in the order of $6 per page on the average.

This author writes about the possibility of translation by machine, but in so doing he makes an excellent case for a very thorough study of all the available literature both in English and in other languages by means of a concrete instance which he quotes. This is taken from what is stated to be the new field of design of electrical switching networks. He points out that anyone looking through the literature would certainly find the pioneer paper in 1938 by Claude Shannon, but that many people who cannot read Russian would miss another article, and that, even if an English translation had been available, the title —*The Application of Boolean Matrix Algebra to the Analysis and Synthesis of Relay Contact Networks*— was not such as to reveal the fact that, next to Shannon's original paper, it was the most important contribution in the field. Locke goes on to calculate that ignorance of the article cost the companies interested not less than $200,000, not to speak of the five-year delay in certain switching-circuit developments.

The librarians at ICI therefore find it necessary to prepare research notes, bulletins, and patents lists. Good abstracts are essential.

In 1955, 72,000 articles on chemistry were considered sufficiently important to be summarised in *Chemical Abstracts*, published by the American Chemical Society.

These facts and figures give some idea of the size of the problem with which librarians and information officers have to deal. Presumably, if there were enough of them, and they worked hard enough for long enough, all the publications would be classified, abstracted, annotated, and stored or circulated, and so brought to the notice of at least some more potential readers. But is this sufficient? Surely not. Merely to present this mass of information even if predigested by excellent abstracts would be to ensure that much of it was wasted. It must be passed to those who can make good use of it.

The librarian–information officer must study the general nature of the problem and of his varied customers, and it is interesting to note what he and his fellow educationalists have recorded.

Aslib (Association of Special Libraries and Information Bureaux) is a non-profitmaking organisation sponsored by the Department of Scientific and Industrial Research. It provides industry with a unique information service, with access to sources of information on any subject in this country and abroad. The following items, as indeed are many in this paper, are abstracted from *Aslib Proceedings*.

At the 1946 Conference of Aslib Dr. R. S. Hutton [13] presented a paper on *The Communication of Specialist Information to Business Executives—The Practical and the Scholastic Attitude*. The problem of bridging the gap between the academic world and the so called practical man dates back a long time. Richard Watson, at that time Professor of Chemistry at Cambridge, wrote in his Chemical Essays in 1781—

> It is not enough to employ operators in this business, a man must blacken his own hands with charcoal, he must sweat over the furnace and inhale many a noxious vapour before he can become a chemist. On the other hand, the practical men themselves are generally illiterate, timid and bigoted to particular modes of carrying on their respective operations. Being unacquainted with the learned, or modern languages, they seldom know anything of new discoveries, or of the methods of working practised in other countries . . . they are unwilling to incur the least expense in making trials, which are uncertain with respect to profit . . . they acquire a certain opinionativeness which effectually hinders them from making improvements, by departing from the ancient traditionary precepts of their art.

Lyon Playfair in his Introductory Address at the Royal School of Mines in 1851 said that—

> If England is to keep pace with other countries as a manufacturing nation, it must be by her sons of industry becoming humble disciples of science. At present her reliance in the "practical" or "common" sense of

her population is the sunken rock directly in the course both of her agriculture and manufactures . . . Depend upon it, an empirical knowledge of practice is not the way now to succeed in the struggle of individuals, or in the struggle of nations. Intellect is on the stretch to get forward, and that nation which holds not by it will soon be left behind. For a long time, practice, standing still in the pride of empiricism and in the ungrateful forgetfulness of what science has done in its development, reared upon its portal the old and vulgar adage, "an ounce of practice is worth a ton of theory".

One wonders whether any of this criticism still holds.

Later Dr. Hutton says: "A practical man has been defined as 'a man who practises the errors of his forefathers'."

He also deals faithfully with the specialist (knowing more and more about less and less), "those who have wasted their time at University by devoting an exaggerated amount of time to social activities and sport", the contrasting "self-made" man (one is reputed to have said: "The dafter I looks, the better I does"). Of the business executive, Dr. Hutton says—

He has necessarily to be a jack of all trades even sometimes at the expense of being master of none. His task is to correlate and assemble the many diverse factors of a business problem. He must have the faculty of rapidly switching his attention from one subject to another, one of the most fatiguing mental operations when actively pursued during the full working day. He does not have time to read and therefore has an aversion to specialist literature.

Later he says that the executive "wants one specific thought and he wants it boiled to the bone and available instantly".

Here is another view of the business man [14]—

Business men are the true barbarians of the modern world. They do not learn. Possibly something could be done with them if positive action could be taken as regards spreading the appropriate documents.

It is easy to understand how one authority specified the ideal information officer as possessing "a lot of common sense, an inquiring mind, a high interest in other people, and a vast amount of tact".

SELECTION

Having attempted to assess both the nature of the problem and some of the difficulties which confront those whose task it is to help us to solve it, how can we best co-operate with them to deal with the flood of papers, journals, reports, pattern cards, and pamphlets with which we are all inundated? Surely a definite system must be evolved by each individual based upon ruthless selectivity. No one can read everything. At Droylsden we have a system of "first readers", who mark a slip of paper attached to the front of the journal with page numbers as indications to others of articles and items in which they are likely to be interested. This slip is a list of those who regularly read the particular journal. Each individual indicates to the librarian those journals which he wishes to see regularly. I for one mentally classify those which are passed to me into two groups— those which are likely to contain articles I must read thoroughly and those which contain material in which I am not so immediately interested. Five minutes spent at work will show if any particular journal contains anything of sufficient interest to warrant taking it home. Having made the selection, I find it important to try and keep the process of reading fairly continuous. Nothing is more over-facing than a too large pile of unread journals and reports. Time must be found for systematic reading. Odd periods devoted to it are futile and frustrating. It is necessary to have favourable conditions, complete quiet, and a comfortable (but not too comfortable) chair. Having secured these conditions, it is surprising how much one can get through in one or two hours.

Obviously, only the larger organisations will have a full-time librarian, but it is absolutely essential to have someone in charge of books and journals who has not only the time but the interest to see that papers are circulated. In an Aslib article [15] dealing with collaboration between librarians, it is stated that liberal policies are generally followed, but "where, however, the library is still in the state of being run by a director, research manager or the like with the part-time services of a girl who shares secretarial typing or switchboard duties with that of looking after books" a "more short-sighted policy is likely to obtain". I have heard of instances in which this is the case internally, when journals stay on an office desk or a laboratory bench, probably unread by anyone and certainly not passed out to those who could most benefit by reading them.

QUALITY OF WRITING

So far we have dealt with the problem from the reader's angle. What about authors? Why is so much technical material so badly written that, even if one is vitally interested in the subject of an article, reading it may be indeed "a weariness of the flesh"?

Natural ability to write clearly and well even about non-technical subjects is given to few of us, and its study and cultivation must seem an unnecessary and boring additional task to some of those who could most benefit others by acquiring it.

In the preface to *Plain Words* [16] Sir Ernest Gowers says—

. . . It is concerned particularly with the use of English by officials. I do not of course suggest that the principles governing the use of English for official purposes are different from those that govern its use for any other purpose. But there are some faults to which official writing is specially prone and others from which it is comparatively free.

I submit that this is as true of scientific and technical English as of official English.

Length

Excessive length for the purpose is one grave fault. Verbose and turgid writing may be done just to fill a given number of pages, although, from reading old journals and comparing them with those of the present time, it would seem that plain "scissors and paste", "penny-a-line" journalism is less common than it used to be. Some trade papers, nevertheless, publish very pedestrian material and obvious rehashes. This criticism does not mean that a certain amount of reiteration is not desirable. Good summaries are invaluable,

but who is to do them? To quote from an Aslib publication [17]—

> Addison once wrote "when writers have the least opportunities of knowing the truth they are in the best disposition to tell it" which being interpreted for our present purpose means that it is much easier to obtain summaries from the young and inexperienced than from the senior investigator, yet it is the survey combining imagination with ripe judgment that best serves our ends.

It is up to editors and referees to keep repetition within reasonable bounds. Long-windedness, however, may be deliberate and calculated to impress [18]—

> Personally I feel that a wrong ideal is often the cause of much of the trouble. The young man wants a long paper, suspecting, in many cases rightly, that when it comes to applying for a higher post his achievement will be estimated by the number of pages he has produced. He wants one that looks as formidable as possible, judging, in many cases rightly, that the senior who looks at it will think more highly of him if it is full of forbidding phrases and long words, especially if they are new words, such as, let us say, diclastically disintegrated to mean "cut into two bits".

It is recorded that when young authors are asked to say simply what they mean they usually manage to do so quite well. Further, L. J. F. Brimble [19] makes the point that—

> if a potential contributor to *Nature* knows that his contribution will be accepted for publication on condition that he reduces its length to a half or two-thirds, then in more than 95% of the cases the necessary reduction is made by the author himself.

Here are two examples from recent articles which I do not propose to identify—

(1) In this first one I have merely italicised words which, I suggest, are not necessary—

> Direct Labour Defined: *It is desirable that the definition of direct labour be fully appreciated at the outset.* Direct labour is that which is directly concerned with the manufacture of the product—or, in a service industry, the labour concerned with the processing of other firms' products—and whose cost should be related to it. *The meaning of the term direct labour is more fully understood when it is considered in conjunction with that of indirect labour.* Indirect labour is, *as its name implies*, work of an auxiliary nature, aiding the direct labour in such ways as plant maintenance, supervision, materials handling.

By cutting out 41 of the 103 words originally used, I think that the passage has been made clearer.

(2) The next example contains 219 words—

> An important factor in management accounting is the "periodicity of reportage" most suitable for management's needs. No hard and fast rule, applicable to a wide variety of cases, can be laid down; the specific circumstances of each particular case call for individual treatment. A fundamental precept, however, is that the reportage should be essentially short-term. This may be emphasised by pointing to the necessity—as a means for the rapid detection of unwanted trends and costs—of short-term reportage, concerning planned sales, planned profit and planned costs. If the matter is considered from the viewpoint of accounting history, the gradual reduction in control-periodicity over the years is apparent. Four decades ago, the annual profit and loss account was the main feature of company accounting. Progressively, since then, half-yearly accounts have been introduced and have been followed in turn by monthly estimated profit and loss accounts. That the latter were used is evidence of a growing desire, on the part of managements, for better and quicker information on the outcome of their operations. Nowadays, in such sectors of total cost as those relating to direct labour and product materials, it is not uncommon in manufacturing industries to find that the periodicity of reportage has been brought down to a daily basis.

I suggest that it can profitably be reduced to 87 words, thus—

> The frequency with which managers should be presented by the accountant with reports regarding costs, sales, and profits will vary considerably, so no hard-and-fast rule can be made. The tendency, however, is for more frequent reports to be required. Forty years ago the annual profit-and-loss account was the main feature of company accounting. Since then, first half-yearly then monthly, and now sometimes weekly or even daily reports are considered necessary by some businesses, particularly on the use of materials and direct labour.

(3) The next example uses a number of high-sounding scientific words and phrases—

> Various investigators have examined the mechanisms of fabric soiling which may be summarised as follows as they apply to carpet soiling:
>
> (1) Mechanisms of impingement and retention (5) are operative in the soiling of textile fibres. By impingement, soil is brought into contact with the fiber through direct transfer and deposition as well as by inertial and diffusional movement of air-borne soil.
>
> (2) The mechanisms of retention (2) involve (*a*) macro-occlusion, or entrapment of particles in the intrayarn or interyarn spaces—filament denier size is a factor in interyarn entrapments and (*b*) micro-occlusion, or entrapment of particles in the irregularities of the fiber surface.
>
> (3) Oil bonding (3, 15) is another important mechanism of soiling which ascribes the fixation of soil to a fiber through adhesion. The presence of oily fixative materials on carpet fibers is due to fiber processing lubricants, contaminants and oils wicked from base fibers.
>
> (4) Electrostatic effects (13) have been mentioned as contributing to soiling although no conclusive evidence has been brought forward to prove the relationship to carpet soiling.
>
> (5) Available surface area (6) is a criteria [*sic*] of soiling and is related to the denier and cross-sectional shape of the fiber.

The meaning of the above 165 words can be more clearly conveyed by 110, mainly shorter and simple words, as follows—

> (1) Carpets become soiled mainly because dirt is trodden into them, although some dirt may be air-borne.
>
> (2) The dirt lodges (*a*) within or between filaments or threads (yarn and filament deniers are factors which influence soiling, as they govern the available surface area) and (*b*) irregularities within the fibre surface. The cross-sectional shape of the fibre is important in the latter case.
>
> (3) Oily materials may contaminate the fibres, accidentally, as added lubricants, or by wicking from base fibres. These fix the dirt, causing it to stick to the fibres.
>
> (4) Electrostatic effects have been mentioned as contributing to soiling, although no conclusive evidence has been brought forward to prove the relation to carpet soiling.

I am prepared to admit that criticism of this kind may be unfair. To procure examples of the type required, it is necessary to take them from their context, and it may well be that some readers find the original statements to be more informative than the paraphrases which I have produced.

In any case, in a report it is often necessary to state the obvious in clear and precise terms, and this may lead to misunderstandings. An amusing

example was discussed in a letter printed in the *Manchester Guardian* [20]—

> The National Institute for Research in Dairying has recently solemnly reported doing much computational work arising out of a large experiment on the spreading properties of butter. "A preliminary factor analysis of the data has been done", we read, "and it is evident that temperature is by far the greatest influence in spreadability".
>
> Was it really necessary, when we are almost daily told of the nation's shortage of scientific manpower, to make a large experiment to discover this? Could not the physicist concerned have saved himself a lot of time in this instance by asking his mum?

The *Manchester Guardian* had some correspondence on scientific English a short time ago, and the scientists had some vigorous defenders. Thus a Mr. Cullum [21] wrote—

> Generally speaking scientists write clear and precise English, because they have to. If a writer fails to express his ideas clearly it is nearly always because he himself does not clearly understand them and this is equally true of scientists and non-scientists— and equally deplorable. Take any scientific journal at random and you will find the quality of its English (clarity and lack of ambiguity being taken as the main criteria) to be very much better than that of the average "business letter" for example . . . If scientific English is the stumbling-block then the fault and its remedy lie with the reader. Scientific English differs from layman's English only in its vocabulary. No one would think it reasonable to read the *Manchester Guardian* without a sufficient vocabulary to make it intelligible; nor is it reasonable to read scientific literature without a sufficient vocabulary. A writer is not guilty of perpetrating bad English if he fails to cater for readers illiterate in his subject.

THE WORK OF THE SOCIETY OF DYERS AND COLOURISTS

No lecture on technical literature presented to dyers would be complete without some mention of the work of the Society in this field, mainly in the *Journal*, the *Colour Index*, the Symposia, Fastness Reports, and textbooks. This will be well known to readers, and the full discussion given at the lectures need not be repeated in the printed form of the paper.

UNIVERSAL DECIMAL CLASSIFICATION

Classification is one of the major problems of the librarian–information officer. Anyone who has tried to arrange personal files satisfactorily soon realises under how many different headings material can be placed. Useful as the alphabetical classification is, it groups "lead pipes" with "Lead Kindly Light". For the individual or small group there seems no doubt that simplicity with plenty of cross-references is the best way out. For the librarian with many books in his care it was necessary to evolve a system which would bring together related concepts and groups of concepts. In 1873 Melvil Dewey, of Amherst College, Massachusetts, U.S.A., first put into operation his "Decimal Classification and Relativ Index". This has developed with international affiliations into the Universal Decimal Classification [22].

It has its opponents: it can be cumbersome and complicated. But it does work. I asked a professional librarian to classify the *Third Report of the Fastness Tests Co-ordinating Committee*, and he did so immediately.

It is—

667.2/3:620.193

667	Dyeing and bleaching industries
667.2	Dyeing
667.3	Textile printing
620	General questions
620.1	Testing materials. Faults in materials
620.19	Defects and deterioration in materials. Corrosion and erosion
620.193	Defects due to physical or chemical action. Resistance to attack.

He summarised the number as meaning: dyeing and printing of textiles from the point of view of resistance to attack by physical-chemical agencies. I think it is pretty good.

COURTAULDS LTD.
DROYLSDEN
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(*MS. received 25th April 1957*)

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COMMUNICATIONS

Transfer of Disperse Dyes to Cellulose Acetate during Steaming

E. H. DARUWALLA and V. R. LIMAYE

The work reported by Daruwalla and Turner [1] has been extended to include a study of the transfer of selected aminoanthraquinone and aminoazo disperse dyes to secondary cellulose acetate. The effects of particle size and chemical structure of dye, presence of dispersing agents, and different thickener compositions have been examined for appropriate conditions of application. With the majority of the dyes studied a constant partition of dye between cellulose acetate phase and thickener phase at equilibrium is maintained with increasing proportions of dye in the system until saturation of the cellulose acetate with the dye is reached. Study of binary mixtures of selected dyes indicates that the transfer characteristics of one component are unaffected by the presence of the other. The results are discussed with special reference to quantitative studies by other workers on the behaviour of these dyes during dyeing.

Introduction

Daruwalla and Turner [1] have described an experimental method designed to allow the study of the transfer of disperse and water-soluble dyes from a starch film to a cellulose acetate film during steaming. During their study they observed that a constant distribution of selected disperse dyes between cellulose acetate and starch at equilibrium was maintained with increasing amounts of dye in the system up to the stage where the acetate phase became saturated with the dye. The shape of the isotherm representing the partition of the dye between the two phases was linear up to this saturation. In recent years linear isotherms have been obtained representing the partition of disperse dyes between secondary cellulose acetate and aqueous dyebath during dyeing by Wahl, Arnould, and Simon [2] and by Bird and Manchester [3], and between polyester fibre and dyebath by Schuler and Remington [4]. The linearity of the isotherm has been explained by Stubbs [5] as representing the initial stages of Langmuir adsorption and by Giles [6] as sorption at specific sites in which the number of equal-energy sites does not diminish with progressive sorption.

In the present work, using the technique developed by Daruwalla and Turner [1], the transfer characteristics of selected aminoanthraquinone and aminoazo disperse dyes have been studied under different conditions of application. The effect of changing the chemical potential of a dye in the system on the distribution of the dye between cellulose acetate phase and thickener phase and on the saturation value of the dye in the acetate has been investigated in detail. Dispersing agents which have been shown by Bird and coworkers [7] to have a considerable effect on the rate of dyeing of disperse dyes have been incorporated in the printing paste in order to study their effects on the transfer behaviour of these dyes during steaming.

Summary of Results

(1) The transfer behaviour of disperse dyes from thickener phase to cellulose acetate phase during steaming is closely similar to their transfer characteristics from water to acetate during dyeing.

(2) Addition of dispersing agents other than Dispersol AC and Emulphor EL to the thickener phase does not affect the transfer of the disperse dyes examined. Both Dispersol AC and Emulphor EL decrease the rate of transfer as well as the amount of dye transferred at equilibrium.

(3) Under standard conditions, the proportion of dye transferred at equilibrium from different thickener films increases in the following order: gum tragacanth < carboxymethylcellulose, potato starch, maize starch < sodium alginate.

(4) With the majority of the dyes studied a constant partition of dye between acetate phase and thickener phase at equilibrium is maintained with increasing proportions of dye in the system until saturation of the acetate with the dye takes place, and this distribution is unaffected by changes in the physical state of the crystalline dye used or by changes in the acetate : starch ratio.

(5) The linearity of the isotherm is not affected by addition of different dispersing agents to the starch paste or by using different substances as thickener compositions, but the slope of the isotherm is altered depending on the specific action of the dispersing agent or the thickener on the dye.

(6) With α-aminoanthraquinone, Celliton Blue Extra, and Celliton Blue 3G, the isotherm representing the distribution of dye between the two phases is non-linear.

(7) In the desorption experiments, in which a film of cellulose acetate containing a disperse dye is steamed in contact with a film of plain starch, a starch film containing a dispersing agent, or films of different thickening compositions, a constant partition of the dye at equilibrium is observed between the fibre substance and the thickener phase. Further, in every case the proportion of total dye present in the cellulose acetate film at equilibrium is the same whether the distribution is effected by adsorption or by desorption.

(8) No definite relation has been found to exist between chemical constitution, molecular size, and aqueous solubility of a disperse dye and its rate of transfer, saturation value in acetate, or partition between acetate and thickener phase.

(9) Study of the characteristics of transfer of selected dyes in binary mixtures indicates that the rate of transfer, amount transferred at equilibrium, and the saturation value for acetate of either component is unaffected by the presence of the other. In addition, either component can be transferred to the acetate phase until it reaches its characteristic saturation value even when the acetate film is already saturated with the other dye component.

(10) Comparison of the absorption spectra of Celliton Orange R and Celliton Fast Blue B when present in a film of cellulose acetate (transferred from starch during steaming) and when present in solution in acetone indicates that there is great similarity in the shape of the two curves.

Experimental

MATERIALS

Dyes

In most cases the commercial products were purified by extraction and recrystallisation until the melting point remained constant, as indicated in Table I.

With dyes (2)–(5) a hot aqueous dispersion was repeatedly extracted with benzene, the concentrated solution chromatographed on activated alumina with benzene as eluent, and the appropriate fraction recrystallised three times from benzene and dried at 60°c. Dyes (7)–(9) were treated similarly, except that the eluent was a mixture of benzene and ethanol.

Dyes (12) and (13) were prepared from diazotised *p*-nitroaniline and mono- and di-methylaniline hydrochloride, the precipitated dye being washed with water, dried at 60°c., and crystallised repeatedly from benzene.

Dyes (14)–(17) were separated from dispersing agent by repeated precipitation of a boiling aqueous dispersion with saturated alum solution. A concentrated benzene solution of the dye was chromatographed on activated alumina with benzene as eluent, and the dye recrystallised from benzene.

Thickeners

POTATO STARCH— A high-grade farina of low ash content (0·3% after ignition with concentrated sulphuric acid).

MAIZE STARCH— A sample of good-quality commercial corn starch supplied for textile printing (ash content 0·25%).

GUM TRAGACANTH— A good-quality powdered white tragacanth (ash content 1·27%).

CARBOXYMETHYLCELLULOSE— A good-quality, medium-viscosity, yellowish powder (ash content 12·9%).

SODIUM ALGINATE— Alg-gum HV/KP (Alginate Maton Frères), a high-viscosity product (ash content 20·8%).

Dispersing Agents

The dispersing agents investigated were a chemically pure grade of sodium oleate (BDH) and the commercial grades (without purification) of the following proprietary products—

LISSAPOL C (ICI)— Sodium cetyl oleyl sulphate

LISSAPOL LS (ICI)— Sodium 1-oleyl-4-anisidine-2-sulphonate

DISPERSOL AC (ICI)— Disodium methylenebisnaphthalenesulphonate

DISPERSOL VL (ICI)— Fatty alcohol–ethylene oxide condensate

EMULPHOR EL (BASF)— Castor oil–ethylene oxide condensate.

Fibre Substance

The acetate films were prepared from secondary cellulose acetate, supplied by British Celanese Ltd., as used in the manufacture of normal acetate rayon and had the following characteristics—

Grade	CP_2
Acetyl value	53·7–54·7 (calc. as CH_3COOH)
Moisture content	4–6%
Viscosity	11–15*

* The viscosity was measured at 25°c. on a solution consisting of 6 g. of cellulose acetate flakes in 100 ml. of a mixture of 95 parts of acetone and 5 parts of water by weight, and was expressed as a percentage of the viscosity of glycerol determined in the same apparatus at the same temperature.

TABLE I

Purification of Dyes and Loss during Steaming

No.	Commercial Name	Makers	*Colour Index* No.	Chemical Constitution (substituents)	Method of Purification	Melting point (uncorrected) (°c.)	Total Dye in Starch plus Acetate (mg. per unit area of contact) Before Steaming	After Steaming (12 hr.)
				ANTHRAQUINONES				
(1)	—	—	—	1-Amino-	Xylene	251	0·790	0·785
(2)	Duranol Orange G	ICI	C.I. Disperse Orange 11	1-Amino-2-methyl-	Benzene	205	0·665	0·652
(3)	Duranol Red GN	ICI	C.I. Disperse Red 9	1-*N*-Methylamino-		165	1·020	0·990
(4)	Duranol Red 2B	ICI	C.I. Disperse Red 15	1-Amino-4-hydroxy-		210	0·408	0·406
(5)	Celliton Fast Pink FF3B	BASF	C.I. Disperse Red 11	1:4-Diamino-2-methoxy-		240	0·493	0·500
(6)	Duranol Violet 2R	ICI	C.I. Disperse Violet 1	1:4-Diamino-	Acetone	261	0·542	0·536
(7)	Celliton Fast Blue B	BASF	C.I. Disperse Blue 14	1:4-Bismethylamino-	Benzene + ethanol	220	1·170	1·140
(8)	Celliton Blue Extra	BASF	C.I. Disperse Blue 1	1:4:5:8-Tetra-amino-		*	1·056	1·062
(9)	Celliton Blue 3G	BASF	C.I. Disperse Blue 31	Methylated 1:4:5:8-tetra-amino-		310	0·590	0·592
				AZOBENZENES				
(10)	—	BDH	—	—	Laboratory reagents	68	0·628	Nil
(11)	—	BDH	C.I. Solvent Yellow 1	4-Amino-		124	0·520	0·091
(12)	—	—	—	4-Methylamino-4′-nitro-	Laboratory preparations, benzene	134	0·189	Nil
(13)	—	—	—	4-Dimethylamino-4′-nitro-		234	0·418	0·409
(14)	Dispersol Fast Orange A	ICI	C.I. Disperse Orange 1	4-Phenylamino-4′-nitro-	Alum + benzene	157	0·652	0·489
(15)	Dispersol Fast Orange G	ICI	C.I. Disperse Orange 3	4-Amino-4′-nitro-		214	0·825	0·816
(16)	Dispersol Fast Scarlet B	ICI	C.I. Disperse Red 1	4-(*N*-Ethyl-*N*-β-hydroxyethylamino)-4′-nitro-		161	0·432	0·429
(17)	Dispersol Fast Red R	ICI	C.I. Disperse Red 19	4-(*N N*-Bis-β-hydroxyethylamino)-4′-nitro-		205	0·787	0·782

* The purity was checked by elementary analysis, as the melting point was not sharp enough.

A 15% (wt./vol.) solution of cellulose acetate in acetone was prepared by thoroughly mixing and then allowing to stand overnight. Acetate films were cast from this solution by the same technique as and with similar spreading apparatus to those described by Daruwalla and Turner [1]. The thickness of the film used was 0·0035–0·0037 cm., and the weight of a disc of the film ¾ in. in diameter, when dried to constant weight at 110°c., was 0·01 g.

Dye Suspensions

Fine suspensions of the disperse dye were made from the purified crystalline material by grinding with distilled water in a small grinding apparatus similar to that used in the earlier work, except that stainless-steel rods 3 in. long and ⅛ in. in diameter were used to effect grinding. The method of preparing the standard stock suspension was identical with that used in the earlier work.

PROCEDURE

Preparation of Dye-containing Printing Paste, Spreading of the Paste, and Drying

Details of the technique have already been given by Daruwalla and Turner [1], and this technique has been adopted without essential modification. In experiments in which effects of different thickener compositions were investigated, conditions of preparation of the thickened paste containing the dye were so adjusted that, after spreading and drying of the paste, the thickness of the films containing different thickener compositions remained the same, and the weight of dye per unit area of contact also was the same.

Steaming

Steam generated at a constant rate in a 3-litre flask (A, Fig. 1), which was heated electrically at a constant wattage, passed on either side through ¼-in. diameter bent glass tubes (B, B_1) into 250-ml. flasks (C, C_1), and thence through glass tubes

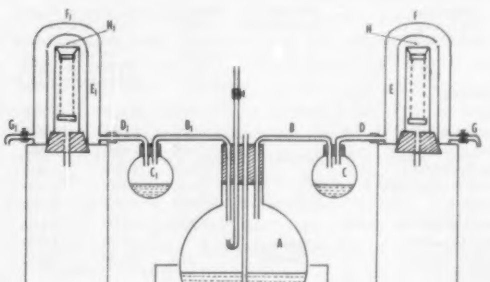


FIG. 1—Steaming Apparatus

(D, D_1) into the outer jackets (E, E_1) of the steaming chambers (F, F_1). This arrangement was found satisfactory in ensuring a constant supply of saturated steam to the steaming chambers and avoiding any condensed water falling on the specimen. Condensed water from E and E_1 was run out continuously through the side-tubes (G, G_1). Constant volumes of water were maintained in the main flask (A) and in the saturators (C, C_1) by separate controls. Before use of the steamer, steam was passed through the apparatus for about an hour so as to ensure steady conditions. The temperature of the inner chambers (H, H_1) remained at 100°c. throughout the steaming period. Introduction of the specimen was carried out as quickly as possible, so as not to disturb appreciably the temperature conditions inside the steamer.

Estimation of Dye transferred

The technique used was the same as that described in the earlier work. In the case of binary mixtures of dyes, the components were so selected that their absorption spectra were completely additive, and no interaction between the two components took place when they were mixed in any proportion. Results are generally expressed as—

$$\text{Exhaustion (\%)} = \frac{\text{(Wt. of dye in acetate)} \times 100}{\text{(Total wt. of dye in system)}}$$

Aqueous Solubility of Dye at 95°c.

The ground suspension (10 ml., concn. 1 g. per litre) was added to 10 ml. of distilled water in a 250-ml. conical flask, which was covered with a long-stem pear-shaped glass bulb. The flask was maintained at 95°c. on a water-bath for 8 hr. with constant stirring for the attainment of equilibrium conditions. The dispersion was then filtered through a sintered-glass crucible (AG 3×4) maintained at 95°c. The filtrate was diluted to 25 ml. with pure pyridine, and the concentration of the dye in the solution was determined colorimetrically.

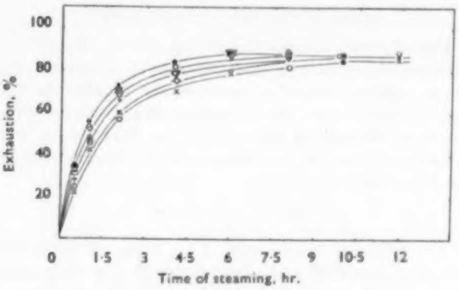
Absorption Spectra of Acetate Film containing Disperse Dye

Different amounts of dye were transferred to acetate films by steaming the latter in contact with starch films containing different proportions of dye for periods until equilibrium conditions were attained. The acetate films were then separated, and the absorption spectra of the dye in the film at different concentrations were determined on a Beckman Model DUV spectrophotometer. The absorption spectra of acetone solutions of the same dye were determined on the same spectrophotometer under identical experimental conditions.

Results

EFFECT OF PARTICLE SIZE OF DYE ON DYE TRANSFER

The purified crystalline dyes (6), (9), (16), and (17) (Table I) were ground for different periods in the rod mill, incorporated in a standard proportion of starch paste, and used for transfer determinations. A similar trend of the results was observed with all four dyes. Fig. 2 shows that, as the time of grinding and hence presumably the degree of comminution increase, the rate of transfer increases, so that the time necessary to reach equilibrium decreases. Direct determinations of particle size have not been attempted, but the results of experiments on the scattering of light by dye dispersions [8] are confirmed by rates of transfer

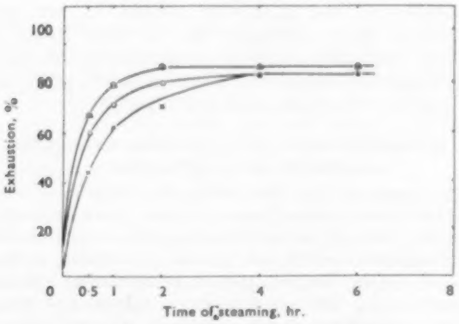


Initial dye content (C) = 0·060 mg. per disc

Time of Grinding

× 10 min. □ 8 hr. ▽ 48 hr.
○ 1 hr. ● 16 hr.
+ 4 hr. △ 24 hr.

FIG. 2— Effect of Grinding upon Transfer of Pure Dispersol Fast Scarlet B from Starch to Cellulose Acetate upon Steaming



Initial dye content (C) = 0·060 mg. per disc

Mode of Preparation of Dye Suspension

× Sublimation △ Grinding for 20 hr.
○ Slow pptn. ▽ Rapid pptn.

FIG. 3— Effect of Particle Size of Pure Duranol Violet 2R on its Transfer from Starch to Cellulose Acetate upon Steaming

in indicating no further increase in fineness after grinding for longer than 16 hr.

In a second series of experiments, the dye used was prepared by different methods, such as sublimation, rapid precipitation by sudden addition of water to an acetone solution of the dye, and slow precipitation by gradual dilution of the acetone solution. Fig. 3 indicates that the method of preparation of the dye, and hence presumably its particle size in the paste, considerably influence the rate of transfer.

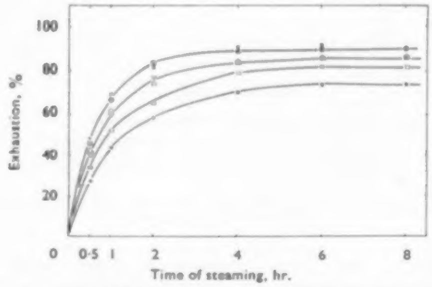
In both sets of experiments it was observed that the same equilibrium exhaustion value was reached if steaming were continued for sufficiently long periods.

LOSS OF DYE DURING STEAMING

Most disperse dyes are likely to sublime on heating and those containing an azo group in the molecule are likely also to be reduced on prolonged steaming. To investigate these possibilities, the total dye content in the cellulose acetate–starch system was determined before and after prolonged steaming. The results in Table I indicate that azobenzene, *p*-aminoazobenzene, Dispersol Fast Orange A, and 4-*N*-methylamino-4′-nitroazobenzene were affected by the conditions of steaming used, and so these compounds were regarded as unsatisfactory for the present investigation.

EFFECT OF DISPERSING AGENTS

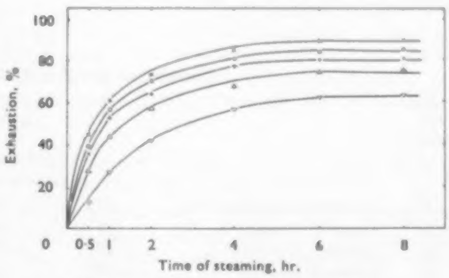
The six products studied represent only a small selection from the great variety of dispersing agents in this field. With sodium oleate, Lissapol C, and Lissapol LS the rate of transfer of Dispersol Fast Scarlet B is increased by increasing the concentration of these agents in the paste (Fig. 4), but in all cases the equilibrium exhaustion remains the same. Addition of Dispersol AC or Emulphor EL results in a decrease in both the rate of transfer and the amount of dye transferred at equilibrium. Emulphor EL has the maximum retarding and restraining action on the transfer, and results obtained with this agent and Dispersol Fast Scarlet B are plotted in Fig. 5.



Initial dye content (C) = 0·060 mg. per disc
Initial Concn. of dispersing agent = 1·0 mg./g. starch paste

● Emulphor EL ▽ Lissapol LS ○ Dispersol VL
□ Dispersol AC × Lissapol C + Sodium oleate
△ Blank

FIG. 4— Effect of Dispersing Agents on Transfer of Pure Dispersol Fast Scarlet B from Starch to Cellulose Acetate upon Steaming



Initial dye content (C) = 0·060 mg. per disc
Initial Concn. of Emulphor EL (mg./g. starch paste)

× Nil △ 1·00
○ 0·25 ▽ 2·00
+ 0·50

FIG. 5— Effect of Emulphor EL on Transfer of Pure Dispersol Fast Scarlet B from Starch to Cellulose Acetate upon Steaming

In a second set of experiments, the effect of additions of all six agents on the transfer of the four dyes (5), (6), (16), and (17) was studied, the concentration of the dispersing agent in the paste being kept the same. With Duranol Violet 2R and

Celliton Pink FF3B, none of the dispersing agents had any appreciable effect.

In general, the effect of additions of dispersing agent on the transfer is more marked with dyes of low aqueous solubility.

EFFECT OF THICKENER COMPOSITION

Results obtained with Celliton Pink FF3B and different thickenings are plotted in Fig. 6. The two aminoazo dyes, Dispersol Fast Scarlet B and Dispersol Fast Red R, were decomposed during steaming when carboxymethylcellulose, sodium alginate, and gum tragacanth were used as thickener compositions. Results indicate that the general transfer behaviour of all the dyes examined is similar when applied from different thickener substances. Sodium alginate is effective in bringing about almost complete transfer to acetate; with carboxymethylcellulose, potato starch, and maize starch exhaustions of the order of 90% are obtained; whereas with gum tragacanth the equilibrium exhaustions are low.

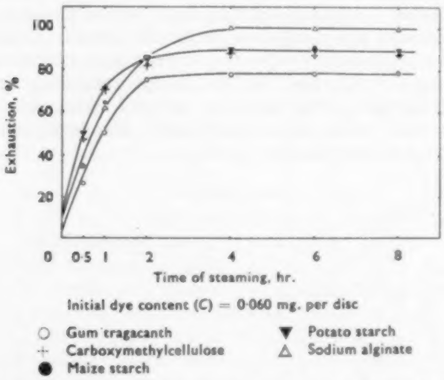


Fig. 6— Effect of Thickener Composition on Transfer of Pure Celliton Pink FF3B upon Steaming

EFFECT OF DYE CONSTITUTION

The rate-of-transfer curves for a number of aminoanthraquinone and aminoazo disperse dyes when applied to cellulose acetate under identical conditions of dye concentration in the starch phase are approximately hyperbolic in form, and by a suitable choice of constants can be represented by the equation—

$$kt = \frac{1}{a - x} - \frac{1}{a}$$

(k = velocity coefficient; t = time of steaming; a = equilibrium exhaustion when the dye transferred to the acetate is expressed as a percentage of the total dye present; and x = the corresponding percentage exhaustion at end of time t). In Table II are given values for the velocity coefficient which give the closest agreement between calculated and experimentally determined rate-of-transfer curves for these dyes.

With aminoazo disperse dyes, the rate of transfer decreases as the molecular weight of the dye increases. Such a relation has not been found to hold in the case of the aminoanthraquinone dyes. The aqueous solubility of a dye does not influence its rate of transfer to cellulose acetate.

DISTRIBUTION OF DYE BETWEEN CELLULOSE ACETATE AND THICKENER

As observed by Daruwalla and Turner [1], with most of the disperse dyes examined, the exhaustion of a dye at equilibrium is independent of the initial concentration of the dye in the starch film until a certain initial concentration is exceeded. From these results an adsorption isotherm has been constructed for each dye, relating the proportion of dye in the fibre substance at equilibrium to the proportion of dye remaining in the starch (Fig. 7).

The following general conclusions apply to the distribution of disperse dyes between the acetate phase and the thickener phase—

(*a*) With all dyes examined except 1-aminoanthraquinone, Celliton Blue Extra, and Celliton

Table II

Molecular Weights and Transfer Behaviour of Dyes

Dye No. (Table I)	Dye	Mol. Wt.	Aqueous Solubility at 95°c. (mg./litre)	Saturation Value in Cellulose Acetate moles per kg. of dry acetate	grams per kg. of dry acetate	Velocity Coefficient $k \times 10^5$	Partition Coefficient*
	Aminoanthraquinones						
(1)	1-Aminoanthraquinone ...	223	35·8	0·048	10·8	129	—
(2)	Duranol Orange G ...	237	14·2	0·052	12·5	59	37·5
(3)	Duranol Red GN	237	32·1	0·084	20·1	313	40
(4)	Duranol Red 2B	239	13·0	0·071	17·0	99	34·0
(5)	Celliton Fast Pink FF3B ...	268	29·0	0·067	17·0	49	46·6
(6)	Duranol Violet 2R ...	238	26·3	0·071	17·1	104	19·0
(7)	Celliton Fast Blue B ...	266	4·75	0·013	3·7	19	5·0
(8)	Celliton Blue Extra ...	268	34·0	0·051	13·9	44	—
(9)	Celliton Blue 3G	332	74·0	0·066	22·1	85	—
	Aminoazobenzenes						
(13)	4-Dimethylamino-4′-nitroazobeneze ...	270	4·05	0·0048	1·3	53	0·76
(15)	Dispersol Fast Orange G ...	242	21·0	0·082	20·0	348	30·0
(16)	Dispersol Fast Scarlet B ...	314	19·5	0·058	18·4	31	22·5
(17)	Dispersol Fast Red R ...	330	31·5	0·034	11·3	26	5·3

* $\frac{\text{Moles of dye per kg. of dry acetate}}{\text{Moles of dye per kg. of dry starch}}$

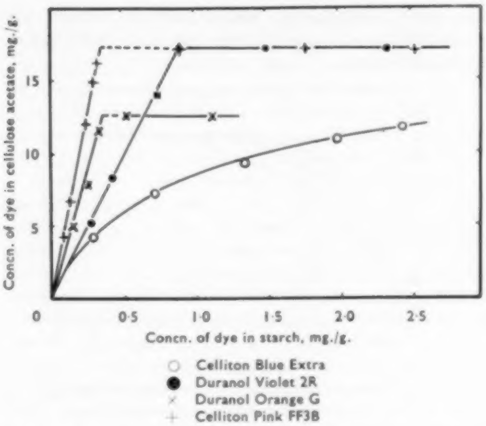


FIG. 7— Isotherms for Distribution of Pure Disperse Dyes between Starch and Cellulose Acetate during Steaming

Blue 3G, over a considerable range of concentrations there is a constant partition between acetate phase and starch phase, which is terminated abruptly when saturation of the acetate is reached.

(*b*) With the three dyes mentioned in (*a*) the isotherm representing the distribution of dye between the two phases is non-linear.

(*c*) Neither the linearity nor the slope of the isotherm representing the equilibrium distribution of Duranol Violet 2R between the two phases is altered when the dye is present in starch in different states of subdivision. For this set of experiments dispersions were made from purified dye obtained by different techniques, such as grinding for 20 hr., sublimation, quick precipitation, and slow precipitation, as mentioned earlier.

(*d*) For the equilibrium distribution of Dispersol Fast Scarlet B between cellulose acetate and starch the isotherm is unaffected by the relative weights of the two phases.

(*e*) Incorporation of different dispersing agents in the starch phase does not affect the linearity of the isotherms for Duranol Orange G, Dispersol Fast Orange G, and Dispersol Fast Scarlet B,

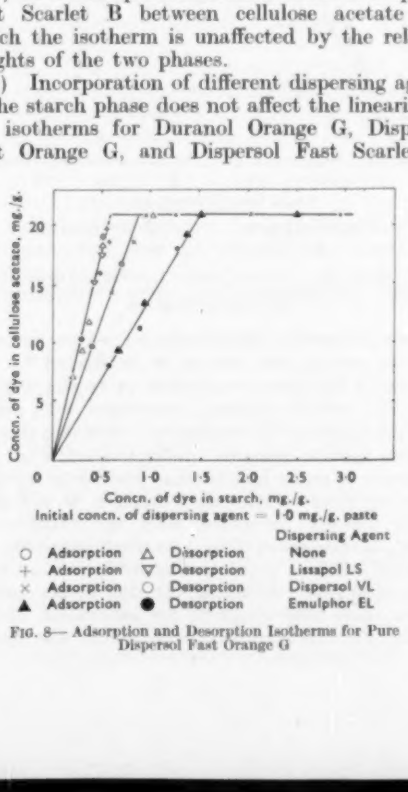


FIG. 8— Adsorption and Desorption Isotherms for Pure Dispersol Fast Orange G

but the slope of the isotherm is altered depending on the specific action of the dispersing agent on the dye (cf. Fig. 8). Amongst all the dispersing agents studied, Emulphor EL appears to be most effective in shifting the equilibrium distribution towards starch with all the three dyes studied.

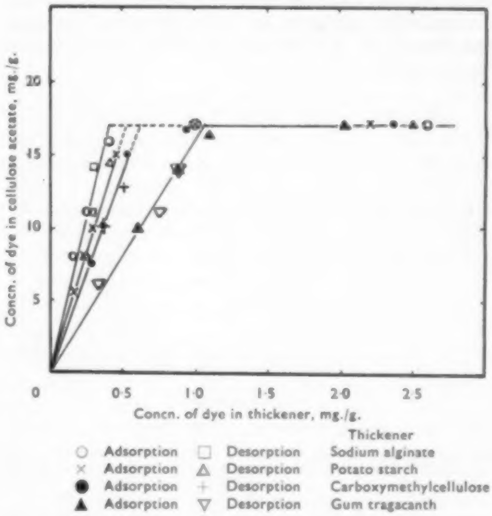


FIG. 9— Adsorption and Desorption Isotherms for Pure Duranol Red 2B

(*f*) With Duranol Red 2B and Duranol Orange G the isotherm remains linear, but its slope is different for different thickeners (cf. Fig. 9). In general, the specific effect of each thickener is the same for both the dyes studied, sodium alginate being effective in shifting the equilibrium distribution of both dyes to the acetate phase, whereas gum tragacanth favours a shift to the thickener phase.

(*g*) With all the dyes studied, the saturation value for a dye remains the same irrespective of particle size of dye, acetate : starch ratio, incorporation of different dispersing agents in the starch, and use of different thickener compositions.

These results have an important bearing on the mechanism of dye transfer, which will be discussed later.

DESORPTION OF DISPERSE DYES FROM CELLULOSE ACETATE TO THICKENER SUBSTANCE

Purified Duranol Orange G, Dispersol Fast Scarlet B, and Celliton Blue 3G were ground for 20 hr., and the dyes introduced into cellulose acetate in different proportions by steaming a film of cellulose acetate in contact with films of starch containing the dyes. On the dye-containing film a layer of plain starch, of starch containing a dispersing agent, or of thickening composition was spread, and this was dried down in air at room temperature. The compound film was then steamed for sufficient periods for equilibrium conditions to be established, and the distribution of the dye in the two phases was determined.

The following are the general conclusions—

(*a*) With all of the above three disperse dyes, percentage desorption at equilibrium is independent

of the initial concentration of the dye in the cellulose acetate.

(*b*) In the case of Duranol Orange G, Dispersol Fast Orange G, Duranol Red 2B, and Dispersol Fast Scarlet B, the desorption isotherm coincides with the adsorption isotherm, and the distribution coefficient for desorption is the same as that for the corresponding adsorption. Results with Dispersol Fast Orange G and Duranol Red 2B are plotted in Fig. 8 and 9.

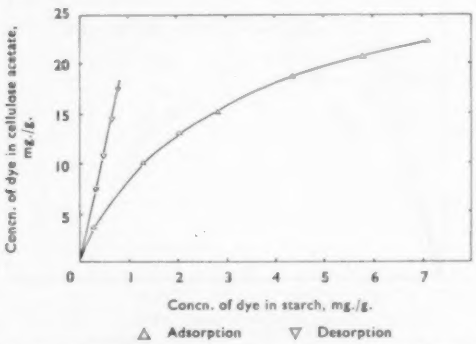


FIG. 10—Adsorption and Desorption Isotherms for Distribution of Pure Celliton Blue 3G between Starch and Cellulose Acetate during Steaming

(*c*) With Celliton Blue 3G, with which the adsorption isotherm is a hyperbola obeying Langmuir's adsorption equation, the desorption isotherm is linear (Fig. 10).

(*d*) Addition of a dispersing agent to starch paste affects the distribution of Duranol Orange G, Dispersol Fast Orange G, and Dispersol Fast Scarlet B between cellulose acetate phase and thickener phase during desorption, depending on the specific effect of the dispersing agent on the dye. However, a linear desorption isotherm is obtained with each dispersing agent, and in each case the adsorption and desorption isotherms coincide. Results with Dispersol Fast Orange G are shown in Fig. 8.

(*e*) When Duranol Red 2B is desorbed from an acetate film, the distribution of dye between the two phases is affected by the type of thickener used, but with all thickener compositions a constant partition of the dye between the two phases is maintained with increasing proportion of dye in the acetate film, and in all cases the desorption isotherm coincides with the adsorption isotherm (Fig. 9).

BINARY MIXTURES

The following three mixtures of disperse dyes were studied—

(21) Duranol Violet 2R and Celliton Fast Blue B
(22) Duranol Violet 2R and Dispersol Fast Scarlet B
(23) Duranol Red GN and Celliton Fast Blue B.

The combinations were so selected that in one case (mixture 21) the two dyes had similar constitutions, in the other (22) very dissimilar constitutions, and in the third case (23) different rates of transfer and different saturation values for cellulose acetate. With each mixture two series of experiments were carried out, the concentration of first one and then the other component being varied, while the concentration of the remaining dye was maintained constant.

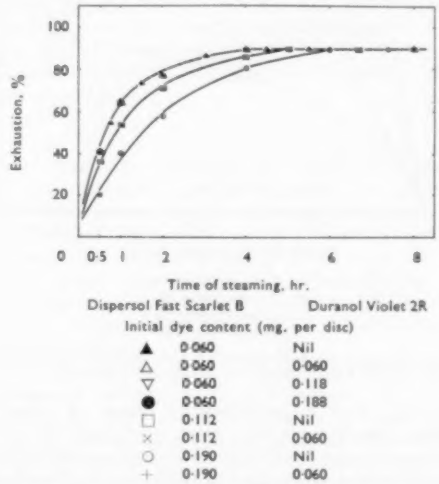


FIG. 11—Transfer of Pure Dispersol Fast Scarlet B in presence of Different Amounts of Pure Duranol Violet 2R from Starch to Cellulose Acetate during Steaming

With all three mixtures, the rate of transfer and the amount of each dye transferred at equilibrium are not affected by the presence of the other component in any proportion (cf. Fig. 11).

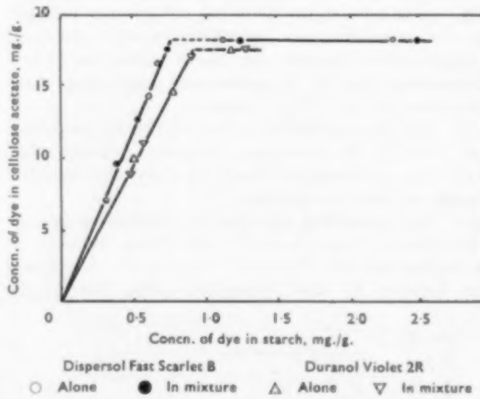


FIG. 12—Binary Mixture of Pure Duranol Violet 2R and Pure Dispersol Fast Scarlet B—Distribution of Dye between Cellulose Acetate and Starch during Steaming

The equilibrium distribution of a dye between cellulose acetate and starch is unaffected by the presence of the other component up to the stage at which the acetate becomes saturated with either dye, and similarly the saturation value of a dye for cellulose acetate remains unaffected (cf. Fig. 12).

In order to study how far it is possible to transfer one component of the dye mixture to cellulose acetate already saturated with the other component, a saturated film was first prepared by steaming a film of cellulose acetate in contact with a film of starch containing the dye in proportion greater than that required for saturation of the acetate. On this saturated acetate film was spread

a layer of starch containing the second dye component in proportion greater than that required for saturation of the acetate, and this was dried down in air at room temperature. The compound film was then steamed for a sufficient length of time to attain equilibrium conditions, and the distribution of each dye in the two phases was determined.

Results indicate that with all the three mixtures studied it is possible to transfer either of the components to its saturation stage in cellulose acetate even when the acetate is already saturated with the other component. During this transfer some of the dye from the acetate containing the dye at its saturation value gets desorbed into the starch phase, but the amount desorbed does not depend on the proportion of the second component going into the acetate phase, and is the same as that desorbed into a plain starch phase.

Discussion

EFFECT OF PARTICLE SIZE OF DYE

The influence of the degree of subdivision of sparingly soluble disperse dyes on the course of dyeing has been studied by Clayton [9], Kartaschoff [10], Vickerstaff and Waters [11], and Bird *et al.*[12], increasing fineness generally resulting in an increase in the rate of transfer of dye to the fibre and consequently an increase in the exhaustion for definite dyeing times short of equilibrium.

Results in the present work are similar to those obtained by Daruwalla and Turner [1], and confirm the importance of the particle size of the dye in the thickener phase. During the film-to-film migration of the dye, capillaries of a pore diameter of only 10 A. are available for diffusion of the dye inside the fibre substance, because of the low swelling capacity of cellulose acetate, and only dye particles approaching a state of molecular dispersion are responsible for the entry into the acetate. This is clearly indicated when the absorption spectra of films containing the dye transferred by the steaming technique are compared with those of very dilute solutions of the dye in a suitable organic solvent in which they are present as nearly true solutions, as indicated by the applicability of the Lambert–Beer law. In Fig. 13 are shown the absorption curves of Celliton Fast Blue B when present in cellulose acetate film and in solution in acetone. The shape of the curves in the two cases is very similar, indicating the existence

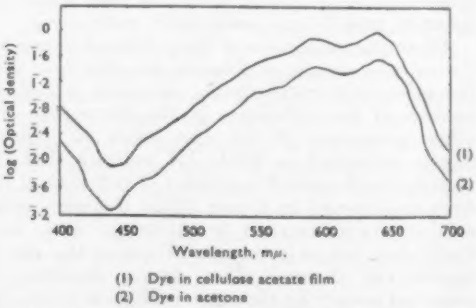


FIG. 13— Absorption Spectra of Celliton Fast Blue B

of a similar state of dispersion in the two media. During the transfer from starch to acetate, as these molecularly dispersed particles are taken up by acetate, further dispersion of the larger particles is induced. The more complete the primary mechanical subdivision, the more readily can the molecularly dispersed particles be furnished for adsorption by the acetate. Results of the present work differ in some respects from those obtained by Vickerstaff and Waters [11], who observed that the equilibrium exhaustion of 1-methylamino-4-anilinoanthraquinone from aqueous suspension at 85°c. increased with time of grinding. This may be explained on the basis of the differences in experimental conditions in the two cases, viz. the more efficient grinding, high temperature of application, specific action of starch, and the high concentration of dye due to the small volume of the starch layer employed in the present work.

EFFECT OF DISPERSING AGENTS

The importance of additions of wetting and dispersing agents to dyebaths for the dyeing of secondary cellulose acetate with disperse dyes has been indicated by many workers, including Corbière [13], Wahl, Arnould, and Simon [2], and Bird and his coworkers [7]. Bird [14] is of the opinion that dyes which are less soluble in water show a reduced rate of dyeing, because dyeing takes place from a saturated aqueous solution of the dye, and therefore very low aqueous solubility acts as a bottleneck in dyeing. He observed that with dyes of low aqueous solubility addition of a dispersing agent to the dyebath resulted in the solubilisation of the dye and subsequently in an increase in the rate of transfer of dye to cellulose acetate; whereas with dyes which have fairly good solubility in water, the dispersing agents stabilised the dye suspensions, and acted as restraining and retarding agents.

In the present work, unlike dyeing, additions of dispersing agents to the thickener phase have a less marked effect on the rate of transfer. It is very likely that, under the conditions of steaming, when a temperature of about 100°c. is attained and the starch phase itself acts as a dispersing agent, the rate of solubilisation of the dye is already quite high and, therefore, additions of dispersing agents do not have any very great effect on the solubilisation of the dye. As has been indicated earlier, the transfer of disperse dyes to cellulose acetate takes place through the diffusion of dye molecules approaching a state of molecular dispersion, and the rate of transfer is mainly governed, therefore, by the rate of dissolution of the dye particles in the thickener. This is substantiated by the fact that, except with Dispersol AC and Emulphor EL, the rate of dye transfer is increased to a varying extent by the presence of dispersing agents in the printing paste, and with increasing proportions of these agents in the paste the rate of transfer is correspondingly increased. Further, the effect of dispersing agents is more marked with dyes of low aqueous solubility, e.g. Dispersol Fast Scarlet B (aqueous solubility 19·5 mg./litre at 95°c.), as compared with those having moderate solubility in water, e.g. Dispersol Fast Red R and Duranol

Violet 2R (aqueous solubilities of 31·5 and 26·3 mg./litre respectively).

With all the dispersing agents studied except Dispersol AC and Emulphor EL, the amount of dye transferred at equilibrium is unaltered by the presence of dispersing agent in the paste. As will be discussed later, the transfer at equilibrium of a disperse dye from starch to cellulose acetate is governed mainly by the partition of the dye between these two phases, depending on its solubility in each phase, and as the experimental conditions used in the present work are such that the dye is present at equilibrium in a state of subdivision approaching that of molecular dispersion in both phases, even in absence of a dispersing agent, additions of these agents will have little effect in changing the chemical potential of the dye in the starch phase.

Dispersol AC and Emulphor EL show both retarding and restraining effects on all the dyes investigated. No definite explanation can be put forward for this behaviour, as these agents have no common feature in their chemical constitution or efficiency of solubilisation for the dye. The dye molecules may get trapped in the micelles of these dispersing agents, and so the amount of molecularly dispersed dye available for diffusion inside cellulose acetate may be considerably reduced, leading to a corresponding decrease in rate of dye transfer, a mechanism similar to that suggested by Speke [15] for the dyeing of vat dyes on cellulose from dyebaths containing Dispersol VL as levelling agent. As the dye molecules are likely to get trapped in the micelles of the dispersing agents, the chemical potential of the dye in the thickener phase will alter, and a corresponding change in the equilibrium distribution of the dye between the acetate phase and the starch phase should take place. This has actually been observed in the present work, the distribution of the dye being shifted to the starch phase when Dispersol VL or Emulphor EL is present in the starch paste (Fig. 8).

EFFECT OF THICKENER COMPOSITION

Reilly and Turner [16] made the striking observation of large differences in the transfer of pure Chlorazol Sky Blue FF (C.I. Direct Blue 1) when transfer was effected from different thickener compositions. Similar results were obtained by Patel and Turner [17] and El Borai and Turner [18] during studies of the transfer of acid and solubilised vat dyes respectively. The differences in the equilibrium absorption values with different thickening agents in the system for constant weights of thickener, substrate, and dye respectively when they are associated with a standard area of contact between the two films have been attributed to differences in the interfacial potential, which are different with different thickeners, to changes in dye concentration in the thickener phase due to differences in the internal structure of the thickener leading to differential water uptake during steaming, and to the attachment between molecules of dye and those of thickener compositions.

In the present work, the equilibrium exhaustion for the disperse dyes studied varied between 72% and 100% when the transfer is effected from different thickeners (Fig. 6). These differences are much less than those observed by Reilly and Turner [16], who showed equilibrium exhaustions for Chlorazol Sky Blue FF of the order of 33% with gum tragacanth and 100% with sodium alginate. For the present study it is reasonable to believe that, in spite of the high surface potential of cellulose acetate [19], interfacial potential differences will not play a prominent part, as the disperse dye molecules carry an infinitesimal electric charge as compared with that on a molecule of Chlorazol Sky Blue FF, which has a high negative charge as indicated by cataphoretic measurements. Further, differences in the water content of the thickener film as a result of using different thickener substances, leading to differences in the concentration of the dye solution in the thickener film, do not appear to play any part in the transfer of these dyes. For instance, although gum tragacanth retains more dye than any other thickener, its equilibrium water uptake is 123·4%, which is intermediate between those of sodium alginate (414%) and of maize starch (94·2%). Further, with carboxymethylcellulose, potato starch, and maize starch the rates of transfer and the equilibrium exhaustions are nearly the same, even though the proportions of water taken up at equilibrium are widely different (274%, 114·2%, and 94·2% respectively).

It appears that the main factor which influences the rate of transfer and the partition at equilibrium is the chemical potential of the dye, which depends on the type of thickening agent used, on account of either attachment of dye molecules to those of the thickener molecules or entrapment of dye molecules in the complex internal structure of the thickener films. Low values for exhaustion with gum tragacanth are rather difficult to explain, as precise data for the film it produces are not available, but with all the dyes examined gum tragacanth has the greatest tendency to keep the dye in the thickener phase, irrespective of the chemical constitution of the dye or its aqueous solubility.

SIMILARITY BETWEEN DYEING AND PRINTING

The high values for equilibrium exhaustion of disperse dyes during dyeing as recorded by other workers agree in general with the observations in the present work. Table III indicates a closer agreement between the results of dyeing with commercial brands and those obtained in the present printing system than has been found for direct [16], acid [17], and solubilised vat [18] dyes.

Although comparison of the published values for the rates of dyeing of selected disperse dyes with the rates of transfer during steaming is difficult because of the differences in the dimensions and other properties of the two kinds of system, results presented in Table III indicate that the velocity coefficients [20] and rates of diffusion of the dyes as obtained in dyeing follow the same order as that during transfer on steaming. It is very likely that, just as in the case of dyeing, the rate of transfer of disperse dyes during steaming is governed mainly by the rate of diffusion of the dye in cellulose acetate.

TABLE III

Comparison of Dyeing and Printing Results

	Dye	Equilibrium Exhaustion (%)		Velocity Coefficient ($k \times 10^6$)		Rates of Diffusion at 80°c.[22] $\frac{ds}{dt} \times d$ (g./sec. per cm.) $\times 10^{11}$
		Dyeing at 85°c. (commercial brands)[20]	From Starch by Steaming (pure dye)	Dyeing with Commercial Dyes[20]	Calc. from Present Work	
(2)	Duranol Orange G	82	91·5	315	59	—
(4)	Duranol Red 2B	83·5	93·0	405	99	0·49
(6)	Duranol Violet 2R	89	88·5	415	104	0·27
(7)	Duranol Brilliant Blue G (Celliton Fast Blue B) ...	95	65·5	67	19	0·036
—	Duranol Red X3B	82	90·3	285	49	0·27
(15)	Dispersol Fast Orange G ...	95	90·5	2500	348	1·81
(16)	Dispersol Fast Scarlet B ...	96	90·3	265	31	0·18
(17)	Dispersol Fast Red R ...	70	66·0	385	26	0·19

From the earlier observations of Green and Saunders[22] and of Ellis[23] a general opinion was formed that increase in the molecular weight of disperse dyes leads to a decrease in the rate of uptake by cellulose acetate. This is supported by comparison of the rates of transfer of Dispersol Fast Orange G, Dispersol Fast Scarlet B, and Dispersol Fast Red R (Table II), with which an increase in the molecular weight of the dye resulting from a change in the substituent group in the dye molecule greatly diminishes the rate of transfer during steaming. The aminoanthraquinone dyes do not show this effect, and so comparisons of this kind are of only limited validity.

Another point of similarity between the two systems is observed when the distribution of a disperse dye between cellulose acetate and dyebath and between acetate and thickener is considered. Wahl *et al.*[2] and Bird and coworkers[3] observed a constant partition of a disperse dye between the acetate phase and the aqueous phase during dyeing; the linear isotherm was not affected by changes in the liquor ratio, and the adsorption and desorption isotherms were identical. Presence of a dispersing agent in the bath did not alter the linearity of the isotherm, but the slope varied depending on the specific action of the dispersing agent on the dye. Similar results have been obtained in the present work also, the constant equilibrium partition being independent of changes in the acetate : starch ratio, etc. (cf. Fig. 7–9).

MECHANISM OF TRANSFER OF DISPERSE DYES

The two main possibilities for the dyeing mechanism on cellulose acetate are solid solution and adsorption of dye molecules on specific sites in the fibre substance leading to equilibrium conditions best defined by Langmuir's isotherm. It may be noted that the distinction between solid solution and adsorption is largely unreal in the case of macromolecules, the only criterion to decide between the two mechanisms being the shape of the distribution isotherm. Experimental evidence is available to support either of these mechanisms. Of late, linear isotherms have been obtained by Wahl *et al.*[2], Bird and coworkers[3], and Majury[24] for the partition of disperse dyes between cellulose acetate and dyebath, and by Schuler and Remington[4] for the distribution of these dyes between polyester fibre and dyebath. Most of these workers prefer to describe the dyeing mechanism as solution of dye in fibre substance, presumably in the non-crystalline regions only.

The present and earlier findings[1] clearly indicate that the transfer of disperse dyes to cellulose acetate during steaming takes place only by means of dye particles whose state of subdivision approaches that of molecular dispersion. Measurements of absorption spectra of these dyes in acetate film indicate that the dye is present in a molecular state of dispersion right up to the stage where saturation of the acetate with the dye takes place. Further, the linearity of the isotherms under all the conditions studied strongly suggests that the dye is similarly dispersed in the starch phase also. It has not been possible to obtain direct evidence of the state of dispersion of the dye in starch, because dye-containing starch films were unsuitable for the measurement of absorption spectra owing to the translucency of the films, and measurements of the dielectric properties of these films could not be carried out on account of the presence of large amounts of water in the film. However, until saturation conditions are reached in the system, the concentration of dye in the starch phase is very low, and under the conditions of the experiments, viz. high temperature (100°c.) and long times of steaming, with starch itself acting as a dispersing medium, the dye in the starch is very likely to be present in a state approaching molecular dispersion.

The effects of dispersing agents and of different thickenings are mainly governed by changes in the chemical potential of the dye in either phase, possibly caused by entrapment of dye molecules in the micelles of e.g. Dispersol VL, Emulphor EL, or gum tragacanth.

With 1-aminoanthraquinone, Celliton Blue Extra, and Celliton Blue 3G the adsorption isotherms obtained are non-linear. However, when the same dyes are desorbed from cellulose acetate film into a plain starch film, a constant partition is obtained irrespective of the proportion of dye in the acetate phase, and the value of the partition coefficient obtained in the desorption experiment is nearly the same as that observed in the initial stages of adsorption (cf. Fig. 10). The curvature of these adsorption isotherms may be attributed to the fact that with these dyes the grinding machinery or the experimental conditions

of steaming do not effect complete breakdown of the crystalline dye particles, and therefore the proportion of primary particles available for diffusion in acetate is considerably less than that which would have been available had grinding and dispersion in the thickener phase been more effective. It is very likely that with suitable dispersing agents or under strong conditions of dispersion practically all the disperse dyes would yield linear isotherms.

One of the most striking observations recorded in the present work is that under all the experimental conditions investigated the saturation value of a dye for cellulose acetate is not affected. Stubbs[5] has suggested that the linear portion of the isotherm represents only the initial stages of Langmuir adsorption, as after a certain stage the solid dye present has a very low chemical potential, only a small fraction of the available sites being occupied, and therefore true saturation of the fibre is not realised in the presence of solid dye. Under the experimental conditions used in the present work, no solid dye is likely to be present in the starch paste at equilibrium with most of the dyes studied, the chemical potential of the dye being altered in some cases either by incorporation of dispersing agents in the starch paste or by using different thickener compositions. If Stubbs' arguments are valid, the saturation value of a dye for acetate in each of these cases should have been different. This is, however, not the case, and therefore it is more appropriate to express the distribution of the dye between the two phases in a manner similar to that of a solute between two immiscible solvents.

Linearity of the isotherm is described by Giles[6] as consistent with conditions under which the number of equal-energy sites does not diminish with progressive sorption. According to him this may be possible where the solute but not the solvent acts as a swelling agent for the substrate, each solute molecule when attached forcing apart molecules of substrate and exposing fresh sites, or where a bombarding solute molecule becomes attached to one of its own kind, previously attached to the substrate, as readily as it would to an unoccupied site in the fibre substance, with the result that a multilayer of solute molecules is readily formed on the fibre substance. Exact data on the swelling of acetate with these simple aminoanthraquinone and aminoazo compounds are not available, but if the first suggestion of Giles is valid, dyes similar in constitution should swell the fibre to the same extent, and consequently the saturation value of the two should be more or less the same. This is not observed in the present work, where the saturation values of two similar dyes, viz. Duranol Violet 2R (1:4-diaminoanthraquinone) and Celliton Fast Blue B (1:4-bismethylaminoanthraquinone) are widely different (17 and 3·7 g./kg. dry acetate respectively). Regarding the second argument, it has been shown from measurements of absorption spectra (Fig. 13) and from the results of desorption experiments (Fig. 8 and 9) that in the acetate film there is no formation of a multimolecular layer of the dye. As will be discussed later, further evidence is available from attempts to introduce a dye into an acetate film containing another dye at its saturation value, the amount of the first dye transferred being the same as when transfer was effected into a plain acetate film. The results of the present work, therefore, indicate that the linear isotherms obtained represent true distribution of a disperse dye between the acetate phase and the starch phase up to the stage of saturation of cellulose acetate with the dye, and are not representations of either the initial stages of Langmuir adsorption or sorption at specific sites of equal energy which do not decrease with progressive sorption.

EFFECT OF DYE CONSTITUTION

As is the case with dyeing[14], the rate of transfer of disperse dyes to cellulose acetate during steaming is independent of the solubility of the dye in water at 95°C. (Table II). These results tend to indicate that the rate of transfer of disperse dyes does not depend on the rate of dissolution of the dye in water or the starch–water phase but is mainly governed by the rate of diffusion of dye molecules inside the fibre substance, which in turn is influenced mainly by the size and the shape of the dye molecule. With aminoazo dyes, increasing the molecular weight of the dye either by introducing bulky nitro groups into the dye molecule or by substituting the hydrogen atoms in the primary amino group generally leads to a decrease in the rate of transfer. In the fine structure of cellulose acetate the capillaries present have a diameter of the order of 10 A. only, and therefore, in the absence of suitable swelling agents, larger dye molecules will find considerable difficulty in diffusing through the fibre substance. In the aminoanthraquinone series, the effect of molecular size is not clearly evident: Duranol Red GN, Duranol Orange G, Duranol Violet 2R, and Duranol Red 2B have very different transfer rates, although their molecular weights are nearly the same. Thus, not only the molecular weight but also the type and the location of groups in the dye molecule play an important part in determining the transfer characteristics of these dyes.

No general correlation has been found to exist between the rate of transfer of a disperse dye into cellulose acetate during steaming and its distribution ratio between acetate and starch. Such a relation is not to be expected, because the velocity coefficient is mainly dependent on the size and the shape of the dye molecule, whilst the partition coefficient is influenced by the relative solubility of the dye in acetate phase and starch phase. With dyes of nearly the same molecular weight, e.g. Duranol Red GN, Duranol Violet 2R, and Duranol Orange G, a relation has been found to exist between the rates of transfer and the saturation values for acetate, dyes having higher velocity coefficients showing higher saturation values.

If Vickerstaff's suggestion[25] of the adsorption of disperse dyes on specific sites in cellulose acetate, by the formation of hydrogen bonds between suitable groups in the dye molecule and the carbonyl group of the ester in the fibre substance, is valid, dyes having groups capable of donating

protons would be readily adsorbed at suitable sites, and consequently the saturation value of these dyes for acetate should be quite appreciable. The results of the present work do not support this type of mechanism. For instance, Celliton Blue Extra (1:4:5:8-tetra-aminoanthraquinone) should have a very high saturation value as compared with 1-aminoanthraquinone or Duranol Violet 2R (1:4-diaminoanthraquinone), but the saturation value obtained in the present work for Celliton Blue Extra is nearly the same as that of 1-aminoanthraquinone, whilst Duranol Violet 2R gives the maximum saturation value amongst these three dyes. Further, in the series of dyes, Duranol Red GN (1-methylaminoanthraquinone), Celliton Fast Blue B (1:4-bismethylaminoanthraquinone), and Celliton Blue 3G (methylated 1:4:5:8-tetra-aminoanthraquinone) the hydrogen atom of some of the amino groups is replaced by a methyl group. Amongst these three dyes, Duranol Red GN shows maximum saturation, Celliton Blue 3G an intermediate value, and Celliton Fast Blue B the smallest saturation value. This, again, is not in agreement with a hydrogen-bonding mechanism, according to which Celliton Blue 3G should show the highest saturation value because of the presence of the large number of proton-donating groups, whereas Duranol Red GN and Celliton Fast Blue B should have more or less similar saturation values on account of internal hydrogen bonding between the carbonyl groups and substituted amino groups. Similarly, in the azo series, there is no indication of the dependence of saturation value of a dye for cellulose acetate on the number of hydrogen-donating groups present therein.

BINARY MIXTURES

During a study of the dyeing of cellulose acetate with binary mixtures of disperse dyes, Vickerstaff and Waters [11] observed that some of the mixtures were additive, each dye component dissolving in the acetate independently, whilst others were non-additive, one dye component interfering with the solubility of the other in acetate owing to the formation of molecular aggregates containing both dyes. Similar behaviour has been recorded during the dyeing of nylon [26, 27], Dacron [28], and Orlon [27] with mixtures of disperse dyes.

If location of dye in the fibre substance is assumed to be on specific sites as suggested by Vickerstaff [25] and by Giles [6], it is very likely that there would be competition between dye molecules for the sites available, with the result that the rate of transfer as well as the saturation value for each component would decrease when it is applied from a mixture containing another dye. The competitive effect would be much more marked in the case of a mixture of dyes of closely related chemical constitutions, as the sites available for adsorption would be the same for both dyes. The results of the present work strongly suggest that the mechanism of transfer of dyes when applied as mixtures can be best explained on the basis of the distribution of each dye component between the cellulose acetate phase and the starch phase depending on its chemical potential in each phase irrespective of the presence of the other component, the only factor of importance being its solubility in each phase rather than the number of sites available for adsorption.

Confirmation of this mechanism is available from the results of the building-up of a dye component on an acetate film containing the other component at its saturation value. With all three mixtures studied it was observed that each component of the binary mixture can be transferred up to its normal saturation value even when the acetate film is already saturated with the other dye component. However, during this transfer a certain amount of the dye from the acetate returns to the starch phase on account of desorption by the starch, but this amount is the same as that observed when an acetate film containing this dye is steamed in contact with plain starch. The presence of the other dye component does not make any difference to the proportion of the first component desorbed. It is also very unlikely that the second dye component gets attached to the first dye component already present in the acetate, because the amount of the second component transferred to cellulose acetate is independent of the presence of the first component and is the same as when transfer is effected on a plain acetate film.

* * *

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Organo-tin Compounds as Textile Preservatives

H. J. HUECK and J. G. A. LUIJTEN

The suitability of a number of organo-tin compounds as preservatives for textiles has been investigated. The investigations comprise tests on the mothproofing and carpet-beetleproofing of wool and on the rot-proofing of jute and cotton. The tests have been combined with experiments on fastness to washing and with leaching experiments.

General Introduction

The work of van der Kerk and Luijten [1, 2] has drawn attention to the highly biocidal activity of certain organo-tin compounds. It appears that these compounds are active against fungi, as well as against insects. It seems therefore worthwhile studying the performance of these compounds in the field of textile preservation, where there is a need for both insecticides and fungicides.

It is well known that wool can be damaged by clothes-moths, carpet-beetles, and a few other insects. Damage to wool by micro-organisms is rare and not of economic importance, at any rate in the temperate zones, where the bulk of wool is used. With cotton and other cellulose-containing textiles, on the other hand, the damage inflicted by micro-organisms is far greater than that caused by insects. In temperate zones the insect damage to cotton textiles in storage may be considered negligible.

An investigation into the suitability of organo-tin compounds as textile preservatives can therefore be limited to two fields of study—(1) the resistance of wool to insect attack and (2) the resistance of cellulose-containing textiles to attack by micro-organisms.

The organo-tin compounds for this study were developed and prepared at the Organisch Chemisch Instituut T.N.O., Utrecht [2, 3].

1. Organo-tin Compounds for the Mothproofing and Carpet-beetleproofing of Wool

INTRODUCTION

A mothproofing compound must be judged first of all as to its intrinsic toxicity towards the insects in question. Its practical usefulness, however, is also determined by secondary factors, among which fastness to washing is of outstanding importance. An investigation into the resistance to repeated washing may even be considered as a standard test in the performance rating of mothproofing agents. The determination of this factor was therefore included in the study of some of the compounds investigated.

METHODS

The compounds to be investigated were dissolved in benzene or trichloroethylene. A suitable sample of a wool serge was soaked with the solution, and pressed until it retained 100% on its dry weight of the solution. In this way concentrations of the preservative of up to 1·0% on the dry weight of the wool were obtained. The tests were carried out in comparison with wool treated with the solvent only.

For the insecticidal tests two methods were employed. In a preliminary series of tests the Stiteler [4] method with visual assessment of damage was used. In later series the Swiss Standard Specifications SNV 95901 and SNV 95902 were followed, which are based on an assessment of the damage by a weight-loss method, test insects being the clothes-moth *Tineola bisselliella* Hum and the carpet-beetle *Anthrenus vorax* Waterhouse. For this purpose circular patterns of wool of known weight, "baited" with yeast suspension, are exposed to attack by larvae of the test insect for 14 days. The weight loss incurred by the patterns treated with an insecticide is expressed as a percentage of the weight loss of untreated test-specimens.

The fastness to washing of the treatment was determined according to the Swiss Standard SVMT 25/A 3011, which entails washing by hand in lukewarm soap suds of specified composition.

RESULTS

The mothproofing capacities of some mono-, di-, tri-, and tetra-alkyltin compounds, tested by the Stiteler method, are shown in Table I. From this it can readily be seen that, of the compounds investigated, the trialkyl compounds, as could be expected, possess the highest activity. In a further series of tests, therefore, further trialkyltin compounds were tested by the Swiss standard method mentioned above. In addition, the fastness to washing of these compounds was determined.

The results shown in Table II are weight losses expressed in terms of percentages of the weight loss of an untreated comparable sample of wool. It is generally accepted that weight losses $< 12\%$ of the blank indicate a satisfactory degree of mothproofing, and that weight losses $< 15\%$ of the blank indicate a satisfactory degree of resistance to the carpet-beetle. For comparison, data on D.D.T. and Mitin FF are included.

DISCUSSION

Tables I and II together give an indication of the relative activity of some trialkyltin compounds in protecting wool from attack by moths and carpet-beetles. Table III gives a list of the compounds classified according to their insecticidal activities.

It is in conformity with the findings of van der Kerk and Luijten [1], who investigated the fungicidal activity of these compounds, that the tributyltin compound has a greater biocidal activity towards these forms of life than the corresponding triethyltin compound.

TABLE I

Mothproofing and Carpet-beetleproofing Activity of Organo-tin Compounds by the Stiteler Test

Compound	Concn. (% on dry wt. of wool)	Resistance to attack* by *Tineola bisselliella*	Resistance to attack* by *Anthrenus vorax*
Tetraethyltin	0·25	—	—
	0·5	+	+
	1·0	+	+
Tetraphenyltin	1·0	—	—
Triethyltin ethoxide	0·05		—
	0·1	+	+
	0·25	++	+++
	0·5	+++	+++
Triethyltin hydroxide	0·1	+	+
	0·25	+++	+++
Triethyltin *p*-toluenesulphonamide	0·1	++	++
	0·2	+++	+++
Triethyltin chloride	1·0	—	—
Tri*iso*propyltin oxide	0·125	—	—
	0·25	+	+
Diethyltin diethoxide	1·0	—	—
Diethyltin dichloride	1·0	—	—
Ethyltin trichloride	1·0	—	—

* Resistance is indicated thus—
— No protection
\+ Slight protection
++ Fair, but insufficient protection
+++ Good protection.

TABLE II

Resistance to Moths and to Carpet-beetles of Wool treated with some Trialkytin Compounds, and the Influence of Washing

Compound	Concn. (%)	Direct Test Weight Loss* *Tineola bisselliella*	Direct Test Weight Loss* *Anthrenus vorax*	Test after Washing No. of Washings	Test after Washing Weight Loss* *Tineola bisselliella*	Test after Washing Weight Loss* *Anthrenus vorax*
Triethyltin hydroxide	0·25	5·7	3·8	1	40	62
				3	52	—
	0·5	1·2	0	1	25	42
				3	34	—
	1·0	0	0	1	12	11
				3	22	—
Tributyltin oxide	0·025	22	39	—	—	—
	0·05	9·3	11	—	—	—
	0·1	0·9	0·2	—	—	—
Diethyloctyltin acetate	0·001	—	101	3	—	101
	0·003	—	77	3	—	98
	0·01	—	57	3	—	105
	0·03	—	60	3	—	106
Diethyllauryltin acetate	0·01	—	72	3	—	106
	0·03	—	56	3	—	103
	0·1	—	21	3	—	120
	0·3	—	3·6	3	—	82
D.D.T.	0·025	18	16	1	93	—
	0·05	4·4	2·9	1	82	—
	0·1	4·2	2·9	1	38	—
	0·25	0·9	0·4	—	—	—
Mitin FF (Gy)	3·0	3·3	2·4	5	3·9	3·6

* Expressed as % of weight loss of the blank.

TABLE III

Classification of some Trialkyltin Compounds according to Mothproofing Activity

Class No.	Compound	Active Concn. (% on dry wt. of wool)
1	Tributyltin oxide	0·05
2	Triethyltin *p*-toluenesulphonamide	0·2
3	Triethyltin hydroxide	0·25
4	Diethyloctyltin acetate	0·3*
5	Diethyllauryltin acetate	0·3
6	Triethyltin ethoxide	0·5
7	Tri*iso*propyltin oxide	>0·25†
8	Triethyltin chloride	>1·0†

* Extrapolated.
† Less active than 6 at indicated concentrations.

Of the four triethyl compounds, three show about the same activity, which is in accordance with the supposition by van der Kerk and Luijten [1] that the intrinsic toxicity is due to the triethyltin ion. Strangely enough, triethyltin chloride does not show any mothproofing activity in the range investigated, though as a fungicide, according to these authors, it is as active as the other triethyltin compounds. The chloride has, however, an appreciable volatility, and may have been lost by evaporation before the insecticidal test was started.

Another discrepancy with the fungicidal activity can be noted with tri*iso*propyltin oxide, which is only slightly active as a mothproofing agent, whereas tri*iso*propyltin compounds, as fungicides, are as active as tributyltin analogues. The data are too few, however, to allow generalisations to be made.

Tributyltin oxide is about as active against *Tineola bisselliella* and *Anthrenus vorax* as is D.D.T. Both compounds are much more active than Mitin FF, if judged solely on their intrinsic toxicity. This compound, however, is far superior to D.D.T. and the best organo-tin compound tested as regards fastness to washing. The organo-tin compounds under discussion, therefore, may be classified as insecticides like D.D.T., suitable for the direct control of clothes-moths and carpet-beetles, but without sufficient permanency. It must be kept in mind, however, that the permanency of Mitin is reached only after a dyeing operation with this substance, involving higher temperatures. With the organo-tin compounds tested, only simple impregnation with solutions at room temperature was carried out; there is therefore the possibility that here also more complex methods of application might lead to satisfactory results.

In this respect, the nature of the fourth group X in the general formula R_3SnX of the trialkyltin compounds seemed to be important. It turned out to be very difficult, however, to find a group X which could not be hydrolysed off easily, so that even in the case of a chemical bond of X with wool, the fastness to washing of the compound R_3SnX–Wool remains doubtful.

Also in some other respects, the mode of action of trialkyltin compounds resembles that of D.D.T. They are equally active against *Tineola bisselliella* and *Anthrenus vorax*. Furthermore, very little insect damage, or none at all, is observed if the concentration of the insecticidal substance on the wool is high enough. It is well known that the toxic action of permanent mothproofing compounds like Mitin, Eulan (FBy), and Lanoc (ICI) appears only after breakdown of the treated wool in the midgut of the larvae. As a consequence, a small amount of damage (though practically negligible) is noticed even in well treated wool. The mode of action of the trialkyltin compounds, therefore, is suggestive of a more direct toxic action like that of D.D.T.

2. Organo-tin Compounds for the Rotproofing of Cellulose-containing Textiles

INTRODUCTION

From investigations carried out earlier [1], it was already known that organo-tin compounds could be used as wood preservatives, in which case they showed remarkable resistance to leaching. Since the compound used (triethyltin hydroxide) is soluble in water, it was concluded that the substance had a special affinity for some component— presumably the lignin— of the wood*.

Resistance to leaching is a very desirable property for textile-rotproofing substances too, so that a closer examination of these substances for that purpose seemed to be appropriate.

At first, experiments were carried out with impregnated jute, because jute contains a lignin compound, making it somewhat comparable with wood. In later experiments the economically more important cotton also was included.

It was found that, under certain conditions, organo-tin compounds are sensitive to light. Because of the extent of the investigation of this factor, the results will be reported separately.

METHODS

The compounds to be investigated were dissolved in an organic solvent like benzene or trichloroethylene. Samples of jute or cotton were soaked with the solution, and pressed until they retained 100% on their dry weight of the solution. The jute used weighed about 350, the cotton 340 g./sq. metre.

Rotproofness was tested by various methods, including a soil burial test, a soil suspension test, and pure-culture mildew tests. Only the results of the soil burial test, the most severe, are recorded in this paper. The results of the other tests showed a similar trend to those obtained by the soil burial test.

The soil burial test was carried out according to the Dutch specification Vitno Bio A1, which in general outline is the same as U.S. Federal Specification CCC–T–191b–5762. The test is performed by burying, for 14 days, strips of the

* Results of experiments on the wood-preserving properties of organo-tin compounds, carried out in co-operation with the Houtinstituut T.N.O., will be published shortly.

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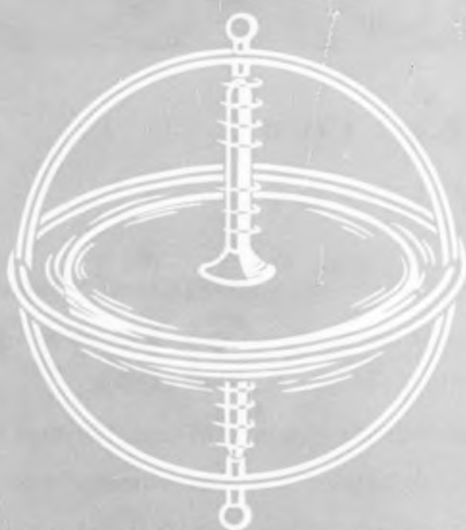

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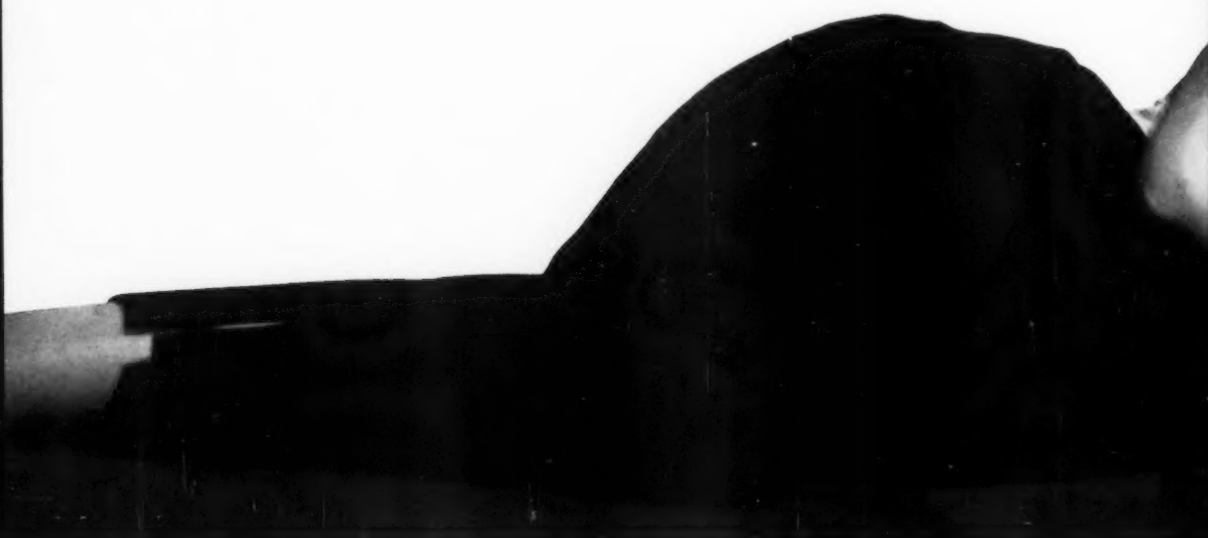

Rationalization in the Wardrobe

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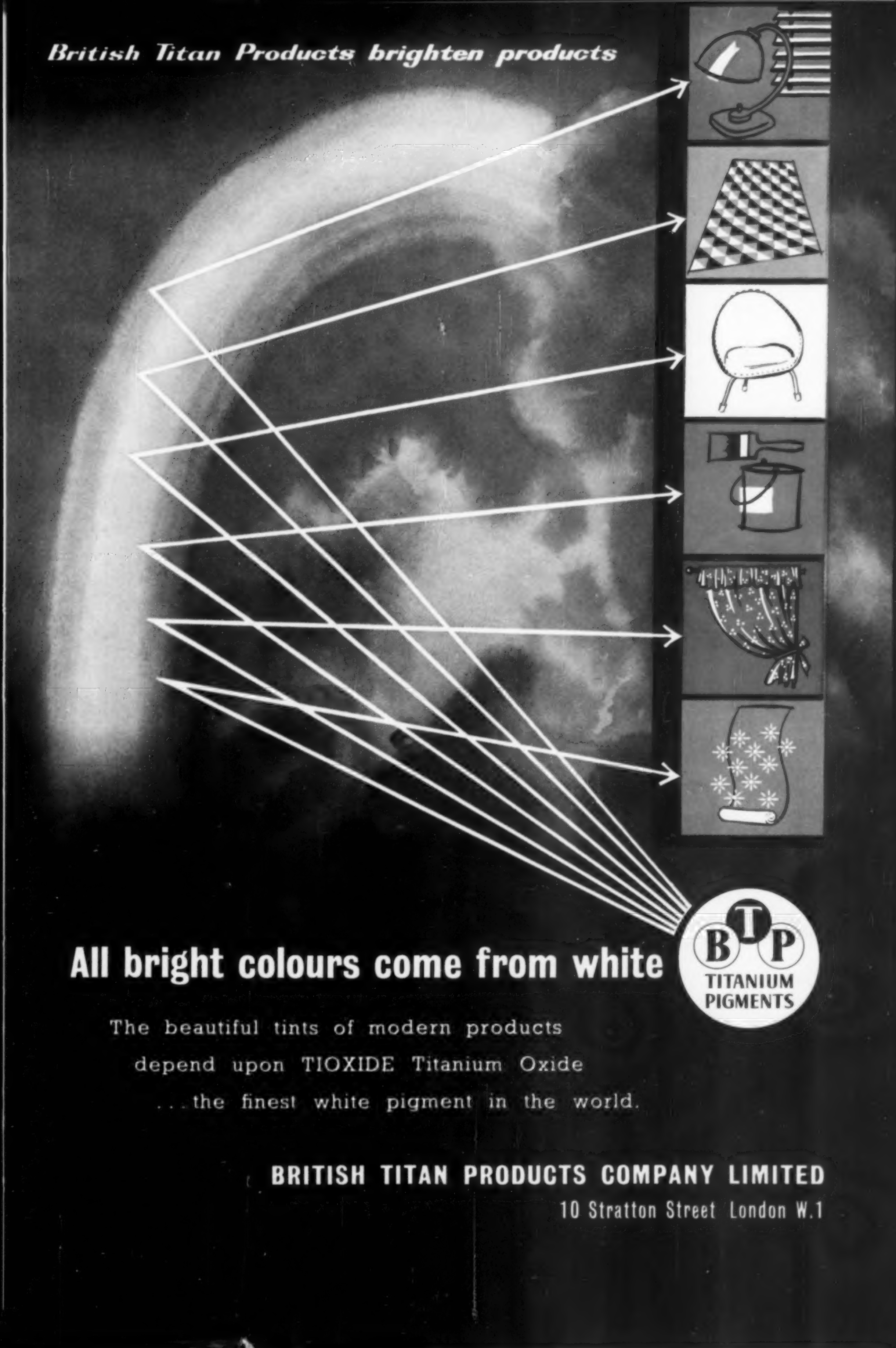
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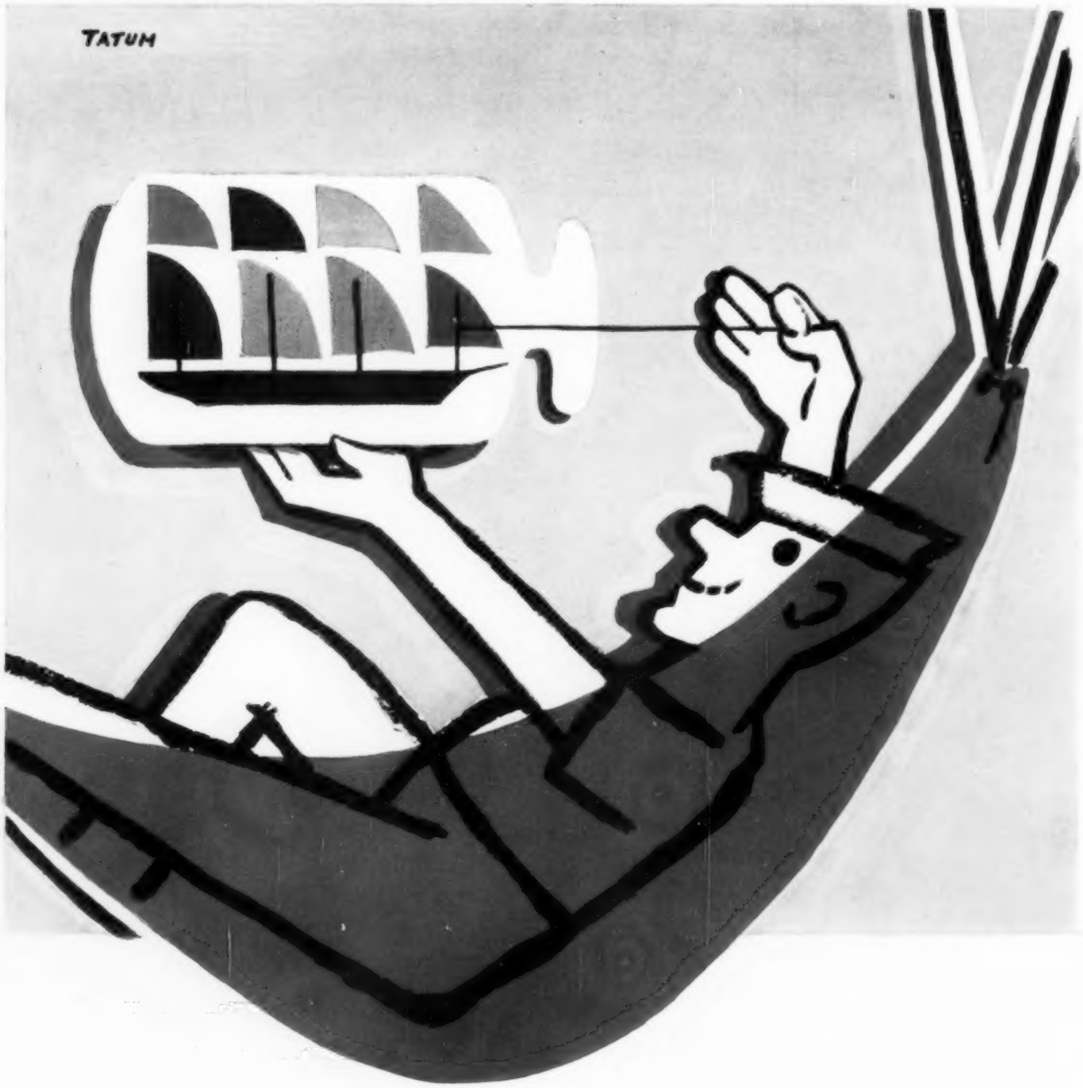
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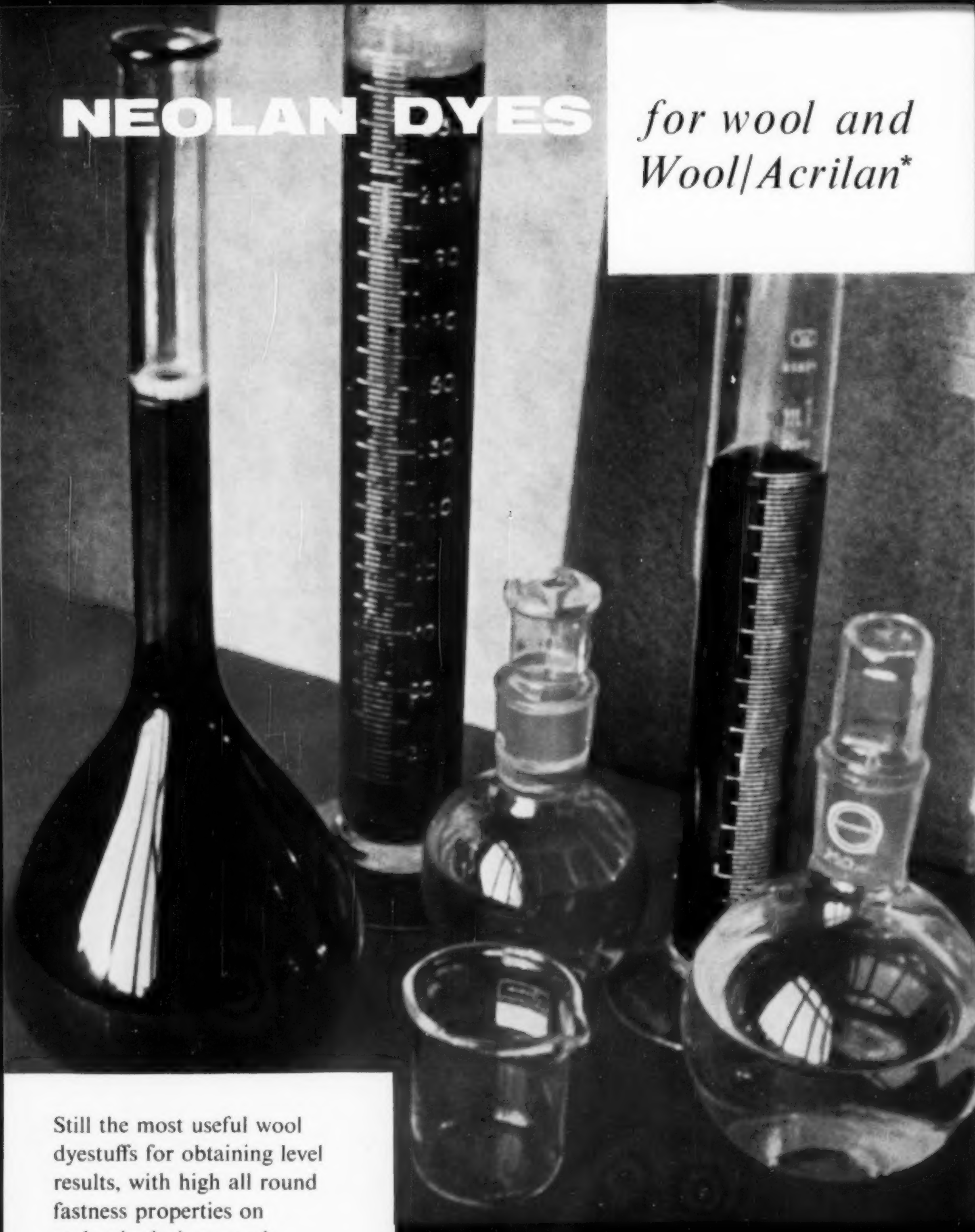
NEOLAN DYES

TABLE IV

Rotproofing Activity of Organo-tin Compounds on Jute and Cotton

Compound	Fabric	Concn. (% on dry wt. of fabric)	Residual Strength (%) Soil burial only	Residual Strength (%) Soil burial after Leaching
Triethyltin hydroxide	Jute	0·034	21	—
Tributyltin oxide	Jute	0·005	1	0
		0·01	7	8
		0·05	65	19
Dimethyloctyltin acetate	Jute	0·01	1	3
		0·03	23	1
	Cotton	0·003	0	0
		0·01	16	25
		0·03	75	56
Dimethyllauryltin acetate	Jute	0·01	7	0
		0·03	11	2
	Cotton	0·003	1	1
		0·01	15	9
		0·03	52	32
Diethyloctyltin acetate	Cotton	0·001	27	48
		0·003	35	26
		0·01	86	71
		0·03	111	78
Diethyllauryltin acetate	Cotton	0·01	26	45
		0·03	50	67
		0·1	77	86
		0·3	95	91
Copper naphthenate	Cotton	0·9	98	91
D.D.M. (2:2-Methylenebis-4-chlorophenol)	Cotton	2·5	111	83
Blank (range of several samples)	Cotton and jute	0–1	0–10	0–5

cotton to be tested in a microbiologically active soil of specified composition. Tensile strength is determined on buried and unburied strips. Most of the samples were leached according to specification ASTM-D 862-45 T-1950, which entails leaching in running tap-water for 36 hr.

RESULTS

The results shown in Table IV are given in terms of residual strengths, expressed as percentages of the initial breaking strength of the fabric. It is generally agreed that a satisfactory rotproofing treatment should ensure a residual strength of at least 90% after a burial period of 14 days.

The results are subject to considerable variation, the magnitude of which shows correlation with the residual strength. At 90% residual strength, the standard error of the mean amounts to 2–4%. At 50% residual strength, this error is much higher.

For the sake of comparison, results with some well known textile preservatives are included.

DISCUSSION

Table IV indicates that especially diethyloctyltin acetate and diethyllauryltin acetate seem to be promising. The first is active even in very low concentrations, comparable with those of mercury-containing fungicides.

Both compounds mentioned appear to possess a considerable resistance to leaching, remarkable because of the very simple method of application used in these experiments. Diethyllauryltin acetate in particular shows this property. Taking into account the possible variation, it seems likely that almost no activity is lost by leaching. With the present fungicides this can be reached only by fixation e.g. with copper, or by application methods involving higher temperatures. The result with diethyllauryltin acetate is suggestive of a special affinity of this compound for cellulose. This type of compound very much deserves to be studied further as to its practical significance.

Two compounds were tested on both cotton and jute, viz. dimethyloctyltin acetate and dimethyllauryltin acetate. In both cases the result with cotton was superior to that with jute. Cotton is a more homogeneous fibre than jute. Taking into account the special affinity of the compounds in question for fibrous substances, it may be assumed that unequal partition between the various components of the jute caused the difference in results.

Conclusions

1. For the mothproofing of wool, tributyltin compounds proved to be as effective as D.D.T. Their fastness to washing was, however, insufficient, so that they cannot yet be used for purposes where permanent mothproofing is required.

2. For the rotproofing of cellulose-containing fabrics, mixed trialkyltin compounds offered much

promise because of their considerable activity, accompanied by fastness to leaching.

* * *

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Application of Azo Pigments to Nylon

Nearly all the practical processes for applying insoluble azo dyes to textiles entail formation of the dyes on the fibre from a coupling component and a diazo component. A number of methods have been proposed for making the azoic dyes *in vitro* and rendering them temporarily soluble by the attachment of suitable groups, which can be removed after application to the fibre. In the present work temporary solubilisation in water has been effected by addition of suitable solvents and alkali. After application to the fibre, removal of the solvent and alkali reconverts the dye to its water-insoluble form. This insolubilisation of the dye is best carried out by washing in water, treating in hot dilute acetic acid, washing in water again, and boiling in detergent.

Owing to the use of caustic alkali, this process is not applicable to alkali-sensitive fibres, e.g. protein, polyester, and some acrylic fibres such as Orlon. Most of the experiments were carried out on nylon, with one on Perlon and one on cotton–viscose rayon.

Para Red is insoluble in cold aqueous alkali, but soluble in alcoholic sodium hydroxide with a violet colour. Bradley[1] used various solvents, particularly pyridine, to prepare solutions of azoic dyes, to which alcoholic or aqueous alkali was added to give an alkali derivative. Water-soluble surface-active agents generally tend to have a solubilising effect on azoic dyes, but the outstanding effect is brought about by cationic agents such as Lissolamine A in alkaline solution. Rowe and Owen[2] solubilised a number of these dyes in water using Lissolamine A and caustic soda, and then carried out dyeing experiments on cotton with these solutions. Their best results were obtained with the azoic dye from Brenthol AS and Fast Scarlet 2G salt (C.I. Azoic Diazo Component 3), but even these were not considered very satisfactory. They also found that β-naphthol pigments gave inferior coloration to those derived from Naphtol AS products.

Six different types of experiment have been undertaken. In the first, which illustrates the general method, a commercial pigment, Monolite Fast Red 2RVS (C.I. Pigment Red 2) was used

TABLE I

Dyebath	A	B	C
1% Aqueous dispersion of Monolite Fast Red 2RVS, c.c.	100	100	100
Industrial methylated spirits, c.c.	500	300	100
Water, c.c.	Nil	200	400
Caustic soda (60°Tw.), c.c.	25	25	25

together with caustic soda and methylated spirits as the solvent. This experiment also shows the importance of suitable amounts of both solvent and water being present to obtain good dyeing results. The pigment, which is supplied by the maker as an aqueous paste, was further diluted with water to give a 1% aqueous dispersion of the commercial pigment. Each of the three dyebaths (Table I) was heated to 60–65°C. and into each was put 2·5 g. light-weight plain-woven nylon material for 15 min. Each piece of nylon was then rinsed in water, soured in hot dilute acetic acid, rinsed in water, and boiled in soap solution, rinsed, and dried. The nylon from dyebath A was dyed a pale colour, the nylon from B was dyed fairly deeply, and that from C was only stained. The amount of dye taken up by the nylon was not measured, nor were the fastness properties determined, but the rubbing fastness was only moderate.

In the second series of experiments a variety of solubilising substances were employed instead of methylated spirits. Monolite Fast Red 2RVS and caustic soda were used as before, each dyebath was heated in a boiling water bath, and the dyed nylon was washed, soured, and soaped as before. Table II

TABLE II

Ethylene glycol	50
Diethylene glycol	30
Ethylene glycol monoethyl ether ...	25
Diethylene oxide	25
Pyridine	15
Brentogen Developer N (ICI) ...	20
Ethylenediamine	25
Morpholine	25
Hexamine	30
Tetraethylammonium hydroxide...	20
Lissolamine A 50% (ICI)	0·3

gives approximate concentrations (%) of the various substances in the aqueous mixture which gave good coloration on nylon.

In the third experiment, nylon and Perlon were dyed together for comparison, a Fast Colour Salt being used to make an azoic dye, and methylated spirits as the solvent. Brenthol AS was coupled with the appropriate amount of Brentamine Fast Blue VB Salt, and the dye made up to an approx. 1% dispersion. A dyebath was prepared as follows—

50 c.c.	Brenthol AS–Fast Blue VB (1% aqueous dispersion)
300 c.c.	Industrial methylated spirits
300 c.c.	Water
30 c.c.	Caustic soda (60°Tw.)

Plain-woven nylon material (2·5 g.) and knitted Perlon (12·5 g.) were dyed in the above solution at 60°C. for 15 min., and then soured in acetic acid and soaped in the usual manner. The nylon was dyed a medium blue, and the Perlon a deep blue.

A fourth experiment was designed to test the stability of an azoic dye containing a *para* nitro group, when used in an alkaline solvent dyebath. Brenthol AS was coupled with diazotised *p*-nitroaniline, and the dye made up to an approx. 1% aqueous dispersion. A dyebath was made up as follows—

100 c.c.	Brenthol AS–PNA (1% aqueous dispersion)
300 c.c.	Industrial methylated spirits
200 c.c.	Water
20 c.c.	Caustic soda (60°Tw.)

Nylon (5 g.), dyed in the above at 60°C. for 30 min., soured in acetic acid, and soaped as usual, was coloured a deep puce instead of the bright red usually associated with this combination. This change from red to a dark blue–red is most likely accounted for by reduction of the nitro group.

A dye was then made from an acetoacetarylamide coupled with a diazo component containing a *para* nitro group. Brenthol AT was coupled with the appropriate amount of Fast Red B salt, and the product filtered, washed, and dried. A dyebath was made up as follows—

0·5 g.	Brenthol AT–Red B powder was ground with
1 c.c.	Diethylene glycol, and heated with
300 c.c.	Industrial methylated spirits and
300 c.c.	N. Caustic soda solution.

Nylon material (2·5 g.), dyed in this bath at 60°C. for 30 min., soured in acetic acid, and soaped as usual, was coloured yellow. In this case there is no drastic change of colour, and the *para* nitro group apparently does not cause instability.

Finally, a benzidine-β-naphthol pigment was prepared, and it was shown to differentiate between cotton and viscose rayon, when Lissolamine A and caustic soda were used for solubilisation. Dichlorobenzidine was tetrazotised and coupled with β-naphthol, and the pigment made up to a 1% dispersion in water. A dyebath was prepared as follows—

12·5 c.c.	Pigment as above (1% dispersion)
62·5 c.c.	Lissolamine A 50% (1% aqueous)
2·5 c.c.	Caustic soda (60°Tw.)
175 c.c.	Water

Cotton–viscose rayon twill lining material (2·5 g.) was dyed in the above at the boil for 45 min. The material was then rinsed, soured in hot dilute acetic acid, rinsed, boiled for 5 min. in 1% Teepol (Shell) solution, and finally rinsed and dried. The cotton was dyed a deep brown, whereas the viscose rayon was only very slightly stained.

Table II is not exhaustive, and isolated experiments have been carried out with other substances, such as acetone and Solution Salt SV (ICI), both of which showed considerable powers of solubilisation. Tetraethylammonium hydroxide was able to act as a combined solvent and alkali, and little improvement was shown on adding caustic soda. It is important that the solvents are stable in boiling alkaline solutions and do not exert a reducing action on the dye. It is also desirable to avoid oxidising agents, as these act fairly readily on the azoic dyes in their solubilised form.

One dye containing a *para* nitro group gives an undesirable result, while another is apparently quite suitable. The suitability of dyes containing nitro groups has not been further investigated.

The last experiment is interesting in that it illustrates a method of distinguishing between cotton and viscose rayon, whereas other similar experiments using benzidine or dianisidine with β-naphthol gave similar results on cotton and viscose rayon. Apparently, under these conditions the particle size of the dye and the dimensions of the pores of the fibres are such that cotton can be penetrated, but not viscose rayon.

Bradley[1] found that dyes from Naphtol AS–G (Brenthol AT) which did not contain a *para* nitro group did not give an alkali derivative and were wholly ketonic in form. With the present technique, however, these dyes follow the general pattern. As an example, the dye from Brenthol AT and Fast Yellow GC shows a solubilising effect with methylated spirits and caustic soda.

In general, it is only necessary to bring together in any order an aqueous dispersion of the dye, a suitable solvent, and an alkali to produce some degree of solubilisation. Solubilising experiments have been carried out with dyes from β-naphthol and arylamides of 3:2-hydroxynaphthoic acid and from arylamides of other *o*-hydroxy-carboxylic acids and aliphatic keto-carboxylic acids and dyes from Developer Z.

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(*27th February 1957*)

[1] Bradley, W., J.S.D.C., **56**, 296 (1940).
[2] Rowe, F. M., and Owen, G., *ibid.*, **52**, 205 (1936).

Notes

Meetings of Council and Committees
May

Council — 7th

Publications — 13th

International Federation 1959 Congress —

Planning and Co-ordinating Committee — 16th

Scientific and Technical Subcommittee — 12th

Perkin Centenary
Dye Statistics and Legislation

The French journal *Teinture et Apprêts* has commemorated the Perkin Centenary with a special issue (No. 36), which, although dated 1956, was not published until November 1957. It is interesting that the cover bears an effigy of St. Maurice, mounted, in armour, reproduced from a seal of the dyers of wool cloth of the fifteenth century, found in the Seine.

The first part of the journal, for which M. Jean Meybeck, Professor of Tinctorial Chemistry in the École Supérieure de Chimie at Mulhouse, is responsible, contains the usual outline of the development of artificial dyes, from the use of picric acid for silk dyeing in Lyons in 1849 and murexide in 1855 up to the modern reactive dyes. In spite of the earlier enterprises, the author feels that Perkin is justly recognised as the inventor of the first synthetic dye. This account is notable for a reproduction of the handwritten practical paper, dated 15th September 1920, in which Professor M. Bader instructed a diploma candidate at Mulhouse, P. Braun, to prepare sulphuric esters of leuco vat dyes, the first representatives of the class of solubilised vat dyes, which Bader developed with his assistant, Sünder.

The second paper deals appropriately with the application laboratories of the dyemaking firms, for it has been stated that Perkin's introduction of the concept of "technical service" was of even greater significance than his discovery of Mauve. The recording of recipes is briefly traced from the Papyrus of Leyden in the third century, through the manuscript of Theophilus in the eleventh and twelfth centuries, to the monumental work on wool dyeing published in the seventeenth century on the instruction of Colbert. Perkin's realisation that the introduction of new dyes demanded the experimental working out of new recipes led in due course to the immense technical service organisations of today, whose varied functions and activities are described and illustrated.

The second part of the special Perkin issue should be of particular value to those concerned with the international trade in dyes. Firstly the definition is given of the term *O.E.E.C. Standard Type*, adopted as a standard of dye strength in statistics published by the Organisation for European Economic Co-operation since 1950. Then follow 24 pages of tables showing production, exports, imports, and apparent consumption of dyes for many countries, all statistics being based on metric tons and French francs for ready comparison.

Two countries— France and the United Kingdom— employ special methods of regulating the importation of dyes, and these are described in detail and compared, the French *décrets* and *arrêtés* of 1938–1950 and the British Dyestuffs (Import Regulation) Acts 1920–1934 being given in full.

Finally, customs nomenclature and tariffs are given for several countries. An international convention on nomenclature for the classification of goods for customs purposes was signed in Brussels in December 1950, and was adopted in 1951 by Germany, Turkey, and the Belgian Congo. This nomenclature was revised by a convention signed in Brussels in 1954, and the revised version has been adopted by France and is being studied by other countries such as Austria, Belgium, the United Kingdom, etc. As far as colorants are concerned, the international nomenclature has adopted a classification based on chemical composition, but it comprises only broad divisions, more detailed classification being left to national legislation.

Second Congress of Colourists in Czechoslovakia

The December 1957 issue of the Textile Edition of the Bulgarian journal *Leka Promishlenost* (**6**, (12), 31–33) contains a report by K. Dimov and S. Serafimov on the second congress of chemist–colourists in the Communist countries of Europe, held in Tatranská Lomnica, in the High Tatra mountains of Slovakia, during 25–28th September 1957. Papers were presented in Czech, German, or Russian. Albania and Yugoslavia were not represented. Of the total of over twenty papers, five were devoted to textile printing, five to leather and fur dyeing, and the remainder to the level dyeing of textiles. The congress was opened by the chairman of the section for chemist–colourists of the Czechoslovak Chemical Society.

On the first day three Czech papers dealt with the high-temperature and carrier dyeing of polyester fibres (Terylene (ICI) or the Czech *Svetlen*), the authors being J. Japoušek of the Textile Finishing Research Institute in Dvůr Králové, Jan Barak of the Wool Research Institute in Brno, and Z. Jelinek from the Research Institute for Organic Synthesis in Roudnice. The third author suggested that certain substances which were cheap and readily available in Czechoslovakia, e.g. solvent naphtha and butyl salicylate, should be used as carriers.

On the second day the congress split into two simultaneous sessions, concerned with level dyeing

and textile printing respectively. To the first topic were devoted four papers, dealing with the tendering by light of vat-dyed cellulosic textiles (by N. I. Abramova, of the Scientific Research Institute for Organic Intermediates and Dyes, Moscow), the ageing of fabrics dyed with Sulphur Black (Z. I. Sergeeva, from the same Research Institute), dyeing problems in the hat industry (L. Štursa and Z. Shmid-Tonak, Nový Jičín, Czechoslovakia), and the continuous bleaching of cotton fabrics (M. G. Shikher, of the Ivanovskiĭ Textile Research Institute, Ivanovo, U.S.S.R.). The first of these papers ascribes tendering to catalysis by the dye of the degradative oxidation of the cellulose by atmospheric oxygen, and gives a method for determining the degree of tendering. In the second paper, the accelerated ageing on cotton is supposed to be due to destruction of the cellulose by sulphuric acid formed from oxidation of disulphides and polysulphides in the Sulphur Black. The Czech authors showed that monosulphonated azo dyes give excellent level results on rabbit-hair felt hats, but the disulphonated dyes are unsuitable. Apart from these four papers, a Russian colleague of Professor Bogoslovskiĭ presented a paper on the disperse dyeing of wool with acid dyes in the form of pigments. Among the acid and mordant dyes are some which give completely insoluble salts with the alkaline earths. The pigments thus obtained are applied as fine suspensions, and are then liberated by treatment with acid (acetic, etc.), giving dyeings of excellent appearance.

The session on textile printing consisted of five papers— on the technical problems of printing with organic pigments (by R. Roup of Dvûr Králové), the soaping of azoic dyeings (K. Janiszowsky, of the Textile Research Institute in Lodz, Poland), recent trends in the design of textile-printing machines (Y. Volan-Tiba, Dvûr Králové), technical and economic problems of calico printing (I. Jakobson, of Warsaw), and the establishment of optimum conditions for dyeing with Variamine Blue B and RT (Jan Lukosz, of Lodz). Dr. Roup examined the difficulties associated with the preparation, preservation, and application of printing pastes containing organic pigments, and outlined the methods worked out in Czechoslovakia for overcoming them. The main difficulties are their relatively rapid drying and their removal from the bowls and runners of the printing machine; other problems are fastness to wet rubbing and the effect on handle. According to the second paper, the main difficulty in "soaping" azoic dyeings is removal of excess of coupling component: an effective method was established using caustic soda and Saponol (α-product, produced by the Polish chemical industry). The aims of recent machine design are improvement of drying following printing, ensuring uniform tension in the fabric, cutting out the runners, and raising the productivity of the machine by combination with continuous agers and washers. The fourth paper compared printing with different types of dyes (pigments, vat, developed) from the points of view of cost and quality. The final paper of the session described a study of the buffering power of various substances with the Variamine Blues, which indicated that the most suitable was zinc chloride, which, added in small quantities to the solutions of Diazols, stabilised them and assisted coupling. Among auxiliary products the non-ionic type were most suitable, as they did not interfere with the reaction producing the dye.

The third day of the congress was devoted to four papers. C. Petzold (Textile Research Institute, Chemnitz, Germany) had investigated the dyeing of polyvinyl cyanide fibres from different sources (Dolan, Dralon, Pan, Prelan, Wolcrylon) with the most generally used dyes and also with certain aftercopperable dyes and dyes applicable above 100°c. He found that the method of dyeing, and the quality of the resulting dyeing, depend greatly on the structure of the polyacrylonitrile fibres, which is not the same for fibres from different sources. O. Mert (Higher Chemical-Technological Institute, Pardubice, Czechoslovakia) found that neither sulphuric nor formic acid, under conditions employed in dyeing, impaired the mechanical properties of Silon (Czechoslovak polyamide fibre). However, that they did influence the fibre was confirmed by determination of the mean degree of polymerisation. A. Lörinc (Budapest) found that the take-up of vat dyes by Perlon was affected by molecular weights of the dyes greater than 900–1000. G. Rösch (Chemnitz) studied various acids, esters, and inorganic acid salts as catalysts in chlorite bleaching, and found acid ammonium phosphate to give the most active and least corrosive bleaching solutions, with minimum evolution of chlorine dioxide and maximum utilisation of chlorite.

The final session comprised five papers. V. Jaszonowicz (Warsaw) reviewed the problems facing the Polish textile-finishing industry— acceleration and simplification of bleaching with reduction in damage to the mechanical properties of the textiles, and working out a method for bleaching synthetic-polymer fibres; methods for dyeing synthetic fibres, dyeing at high temperatures, and application of vat dyes as dispersions; printing with organic pigments, mechanisation of screen printing, and introduction of flock printing; crease-resist finishing, waterproofing, flameproofing, rotproofing, and mechanical finishing ("silk finish", Sanforizing, etc.). Pigment dyeing, especially of man-made fibres, was discussed by K. Bitner (Dye Research Institute, Ústí, Czechoslovakia), who used as binder an epoxide resin having good softness and condensing at the relatively low temperature of 80–90°c., unlike other binders, which condense at 120–145°c. He stressed that the condensation of the resin does not cease on application but continues for some time, which contributes to its keying to the fibre itself. He also emphasised that the handle of the cloth was in no case adversely affected; i.e. no hard film was formed, and the softness of the material remained unchanged. K. Kalin (Bucharest) described the I.S.O. method of light-fastness testing, and stated that it could give results differing by 0·5–1·0 grade. A simplified method

had been worked out. He described also observations on the light fastness of sulphur dyeings subjected to various aftertreatments. T. Robinzon (Žilina, Czechoslovakia) reported on the effect of various finishes on the fastness of direct dyeings on viscose rayon. He found that the fastness depends both on the nature of the dye and also on a whole series of other factors. The final paper of the congress, by R. Roup of Dvůr Králové, described the Czechoslovak Versatin method for applying epoxide resins as binders and thickeners in pigment printing.

On the initiative of the Czechoslovak Section of Chemist–Colourists, it was decided that new sections of colourists should be formed where none exist at present, that such sections should maintain continuous contact for the discussion of problems of mutual concern, and that the sections should organise a congress of this type every two years.

OBITUARY NOTICE

Henry Charles Olpin

We regret to record the death of Harry Olpin, who unexpectedly passed away on 21st March 1958 at the age of 58. A native of Stroud, he received his basic education at Marling School, proceeding to Leeds University in 1919. In the following four years he distinguished himself by obtaining his Dyeing Diploma, together with City & Guilds certificates in cotton, silk, and wool dyeing and in dye intermediates, and was awarded a Second-class Honours Degree in Colour Chemistry, followed by M.Sc. in 1924.

In 1923 he joined the staff of British Celanese Ltd. (with whom he was still associated at the time of his death), becoming one of a group of young men working under G. H. Ellis on the discovery and manufacture of dyes suitable for cellulose acetate. He was appointed Chief Assistant to Mr. Ellis in 1926; he held this post until the death of the latter in 1941, when he was appointed Head of the Dye Research Department in his stead.

A notable achievement of his early days was the introduction and manufacture of S.R.A. Fast Black IV (C.I. Disperse Black 1), which is still the basis of the most satisfactory diazotised-and-developed black for secondary cellulose acetate rayon. A long list of subsequent patents, some 115 of which are abstracted in the *Journal*, stands as a tribute to his ability.

The fact that he had on the wall of his office for many years the following quotation from Colbert (1672), to serve him as an incentive and objective in his work, reveals that he fully appreciated the function of the unit under his control —

> All visible objects are distinguished and recommended by colours, but, for the purposes of commerce, it is not only necessary that they should be beautiful, but that they should be good and that their duration should equal that of the materials they adorn.

He was an assiduous worker for this and other scientific societies, serving on many working committees. He was a member of Council from 1951 to 1954, and served on the Publications Committee from 1953 and the Diplomas Committee from 1952. He took great interest in the work of the Terms and Definitions Committee, of which he had been a member since its inception. He was the first Honorary Secretary of that Committee, and its successful establishment and methods of working owe a great deal to the enthusiasm and energy he displayed during the Committee's early years. Olpin's almost unique industrial experience made him for many years a most valued member of the Burnt-gas Fumes Fastness Subcommittee, and he worked hard on two methods of test that have been proposed as standards.

He also collaborated with several of his colleagues in a number of authoritative papers presented at symposia and ordinary meetings of the Society (J.S.D.C., **67**, 620 (1951); **69**, 357 (1953); **71**, 817 (1955); **73**, 247 (1957)). In 1954 he was one of the first to receive the Fellowship of our Society, to add to a similar honour conferred by the Royal Institute of Chemistry some years previously.

Had he lived a few months more, he would have completed 26 years' continuous service on the Midlands Section Committee. He was several times invited to be nominated as Chairman of the Section, a position which he would have filled with distinction. But owing to a slight disability of speech, of which he was, perhaps, too conscious, he always declined. But he was a tower of strength to successive Chairmen of the Midlands Section.

Harry Olpin was a most likeable personality and is sadly missed by a very large number of people who worked with him either at British Celanese or in the affairs of the Society and who enjoyed his personal friendship. Of a quiet disposition, he was nevertheless a determined and thrustful partisan of any worthwhile cause, and his opinions, backed as they were by mature knowledge and sound judgment, were always respected.

Just before his death he completed an Explanatory Paper on the theory of dyeing with disperse

dyes which he had promised to the Midlands Section. This was actually presented by his colleague, Dr. D. Finlayson, at a most successful meeting held in Loughborough four weeks later.

He is survived by a widow, two daughters, and a son.

A. MELLOR
A. W. CARPENTER

New Books and Publications

Proceedings of the Perkin Centennial

Edited by Howard J. White, Jr. Pp. x + ix + 468. [Lowell, Mass.: American Association of Textile Chemists and Colorists. 1957.] Price, $10.00.

The Perkin centenary or centennial was never in danger of being overlooked during 1956, and the present volume is a complete record of the celebrations, sponsored by the AATCC, held at the Waldorf-Astoria in New York during the week of 10–16th September 1956. After introductory matter giving the organisation of the centennial, the full programme, and details of exhibits, the first group of papers is concerned with colour-fastness testing in various countries— an aftermath of the I.S.O. meeting held in New York during the previous week. This is followed by the address on Perkin given by Sir Robert Robinson, who was Official Representative of The Society of Dyers and Colourists. The rest of the total of some sixty papers were grouped mainly according to different substrates— plastics, cosmetics and drugs, leather, food, paper, textiles— to which colorants are applied. Other groups of papers are concerned with the physics and the psychology of colour, the chemistry of dyes, and the history of dyeing.

This expensively produced volume provides a valuable conspectus of the importance of coloration in our modern life. It is perhaps a comment on our modern technology and economics, even in the U.S.A., that the only colour in this book of homage to colour is in the binding and in a few headings on the preliminary pages, printed in mauve ink.

C.J.W.H.

Historical Notes on the Wet-processing Industry

By Sidney M. Edelstein. Pp. 54. [New York: *American Dyestuff Reporter*. 1957.] No price.

This collection of a dozen papers reprinted from the *Reporter* has been published by that journal in commemoration of the Perkin Centennial 1956. The first paper, on Perkin himself, contains the interesting statement that he discovered an azo dye before he obtained Mauve, though he patented it later. It is followed by a very full bibliography of papers and other writings by and about Perkin. Other papers discuss the early dyers' guilds, Napoleon and the French dyeing industry, Thomas Cooper, Robert Hooke, Chevreul, Home and Black, Yankee dyers, and eighteenth-century printing.

C.J.W.H.

Lehrgang für den Textilveredler

Monthly Supplement to the *SVF-Fachorgan für Textilveredlung*. 1955–. Basle: Schweizerische Vereinigung von Färbereifachleuten. ca. 12 pages per issue. Annual subscription (foreign), 12 Swiss francs.

In January 1955, the S.V.F. commenced the issue, with its journal, of a kind of home educator primarily intended for the younger members of the calling. At the beginning it was proposed that it should continue for three years. This period has passed, and the task set then is less than half completed, so that there seems no justification for further delay in considering this interesting experiment, which should have useful lessons for other technical societies in the same field. Most of them with journals of their own are faced with the same difficult problem. If the journal is a serious organ for the communication of new ideas in appropriate terms, the message it carries for the general membership becomes less effective with each succeeding year. Many members feel that advances in science and technology are passing them by, and that no-one is greatly concerned to help them. A society's interest in the preparation by candidates for its diploma examinations should also make it willing to examine experiments in technical instruction.

The S.V.F. Supplement is a most thorough and extensive effort. The issues are separate, and can therefore fix and maintain the methods of exposition and the standards by which the material is selected, independently of the editorial policy of the journal itself, a procedure which avoids many troubles.

The original scheme divided the whole field of textile knowledge into twenty-one sections, ranging from information about the basic properties of fibres and their manufacture (*Faserkunde*), through the production of yarns and fabrics and their chemical processing, to industrial psychology. Other sections were to deal with topics such as water and effluent treatment, chemistry of fibres, dyes, and auxiliaries, testing and diagnosis of faults, textile machinery, etc.

It is almost a foregone conclusion that an enterprise initiated in Switzerland will be done with the greatest thoroughness, and the present one is no exception to this rule. If it is continued as it has been carried on in its first years, it will represent a collective treatise of information and instruction which will not be equalled by any single textbook available to the textile technologist, young or old. It would be of great value to candidates preparing for our own Associateship examinations, and perhaps even more so for those preparing for the A.T.I.

There are difficulties, of course. One arises from the virtue of thoroughness. Almost the whole of the first year of issue is taken up with *Faserkunde*, and most of the second with yarn and fabric manufacture. Interpolated subjects such as water treatment, coating, and industrial psychology have appeared at random, not necessarily a bad thing,

except that these *are* secondary in the main groups of subjects to which they belong. This random appearance must, however, be disconcerting to, say, a dyer, who may feel that he will have to wait for a year or two before his special interests are dealt with. One does not envy the editor in his task of getting his contributors to send in their considerable efforts on time nor, sometimes, of curbing their enthusiasm. Those who have already contributed have, on the whole, designed their treatments to emphasise information which is significant for the student in industry. They should try to shorten the first treatment of each topic, so that the whole scheme might be covered in a reasonable time. If it takes ten years, this represents undue prolongation of a study scheme, and only generations to come can be satisfied completely. Textile processing, the main interest of the S.V.F., has hardly been touched on yet. Might it not be arranged that a self-contained introductory survey is complete within the next two years? If the *Lehrgang* attracts great support, as it is bound to do, there is the prospect of a corresponding advanced course in the more distant future.

These minor criticisms must not be taken to detract from this sensible and heroic enterprise, and the good wishes of the Society for continuing success should go out to its Swiss counterpart.

H. A. Turner

Marking Containers of Hazardous Chemicals

London: Association of British Chemical Manufacturers. 2nd edition 1958. Pp. v + 108. Price, 10s. 0d. or $2.50.

This manual suggests general principles for the preparation of warning labels. The legal position is first reviewed: apart from specific obligations under various Acts and Regulations, the chemical manufacturer is under a general obligation to give adequate warning and information about any hazardous product of his to his own employees, to carriers, to his customers' employees— to all who are going to handle the chemical up to the time at which the container is emptied for use. It is a purpose of the A.B.C.M. scheme to avoid unnecessarily alarming wording without dilution of the truth. The design and layout should be easily recognisable as a warning label, and the information to be given includes— name of product, signal word (*danger*, *warning*, *poison*, or *caution*), hazard(s), precautions, and accident action and/or first aid. Standard phrases are suggested, to comply with the principle that statements should be brief, accurate, simple, and easily understood.

Six examples of recommended warning labels are given, and recommended wording is set out for some 300 hazardous chemicals, including e.g. acrylonitrile, aniline, *o*-anisidine, *cyclo*hexanol, dimethylformamide, metachrome mordant, 1-naphthylamine, the nitroanilines, and sodium nitrite. About sixty substances were considered but rejected, e.g. because they were not sufficiently hazardous, did not pass commonly in commerce, or were already covered by the Poisons Act and Rules. Among these were hexamine, hydrazine hydrate, potassium permanganate, and tritolyl phosphate.

C.J.W.H.

Proceedings of the Second International Congress of Surface Activity

Edited by J. H. Schulman. London: Butterworth's Scientific Publications (New York: Academic Press Inc.). 1957. Price (four volumes), £16.

I— Gas-Liquid and Liquid-Liquid Interface

Pp. xi + 521. Price, 90s. 0d.

II— Solid-Gas Interface

Pp. viii + 348. Price, 75s. 0d.

III— Electrical Phenomena and Solid-Liquid Interface

Pp. x + 621. Price, 100s. 0d.

IV— Solid-Liquid Interface (Washings, etc.) and Cell-Water Interface

Pp. viii + 352. Price, 75s. 0d.

The fact that a second mammoth congress of surface activity was held in London in April 1956, only two years after the first mammoth congress, held in Paris during August and September 1954, is evidence of the continuing interest taken in the subject and the importance attached to it in modern science and technology. Unlike the proceedings of the first congress (reviewed in J.S.D.C., **72**, 383 (1956)) the present four volumes have been produced by a commercial publishing house (and two printers), who are to be congratulated on bringing out a permanent record of all 200 papers and relevant discussions, clearly printed and properly bound, within a year of the holding of the congress. A minor criticism is that no-one appears to have thought of recording anywhere in these volumes when and where the congress was held; this might be important in dating the papers.

Again, the papers are printed in English, French, or German, and have been contributed from all over the world. Several fall within the field of interest of the Society, and it is hoped to publish abstracts of these later.

The main subject headings in the first volume (presumably relating to different sessions of the congress) are— insoluble films, soluble films, foams, evaporation retardation, monolayers liquid–liquid interface, solubilisation and micelles, emulsions, and general phenomena. One of the papers relates the adsorption properties and the photochemical behaviour of dyes, and another deals with chelation at interfaces.

Volume II is divided into "physical adsorption" and "chemisorption", and includes a paper from the University of Alberta on the thermodynamics of the system wool keratin–water vapour.

The subheadings in Volume III are— contact angles, spreading, and wetting, flotation, adhesion, adsorption, lubrication, and nucleation.

The last volume is probably of the greatest direct interest to readers of this *Journal*, the subheadings being washing, and suspensions and agglomerations. The first section consists mainly of papers concerned with detergents, their analysis, and their

effects on textiles. This volume contains also a paper on the dyeing of a wool keratin derivative in a monolayer. C.J.W.H.

Neueste Fortschritte und Verfahren in der chemischen Technologie der Textilfasern
Zweiter Teil
Neue Verfahren in der Technik der chemischen Veredlung der Textilfasern
Hilfsmittel in der Textilindustrie
Volume III

By Louis Diserens. Pp. xii + 933. Basle and Stuttgart: Verlag Birkhäuser. 1957. Price, Swiss francs or DM 136.00.

Dr. Diserens's *magnum opus*, "Latest Developments and Methods in the Chemical Technology of Textile Fibres", is divided into two parts, each published in three volumes. Part I is called "The Latest Advances in the Application of Dyes", and Part II, "New Developments in the Application of Chemical Finishes to Textile Fibres". The book under review, "Auxiliaries in the Textile Industry", is the last of these six volumes, and incidentally the last of the author's completed works. Before his death in January 1956, he had in fact started an additional volume, and we are told in the preface that others intend to complete the task which Dr. Diserens had set himself.

The present volume deals with four aspects of textile processing and finishing. The first chapter (90 pp.) is a review of methods for increasing fibre strength, particularly in the wet state, thereby promoting dimensional stability. Next comes a chapter (195 pp.) on durable finishes, obtained by both chemical and mechanical methods. Full details are given for the production of permanent chintz and embossed finishes, using synthetic resins. The third chapter (250 pp.) describes the theory and practice of crease-resist finishes, and the last chapter (352 pp.) is a most comprehensive survey of new methods in the field of water-repellent and water-proof finishes. As in the preceding chapters, everything is covered from A to Z.

Each chapter is followed by an impressive table listing relevant commercial products. The tables are set out under the headings— *name of product, manufacturer, chemical composition, literature or patent reference*, and *field of application*, and they represent a most useful feature of the book. Thus, after the chapter dealing with crease-resistance, 85 aminoplast resin precondensates are listed, of which 22 are of German, 50 of U.S.A., 7 of Swiss, 4 of French, and 2 of British origin. Even more comprehensive is the list of products given after the chapter dealing with water-repellent finishes; e.g. 174 products are mentioned as representing the commercially available paraffin or wax emulsions containing aluminium salts.

The remaining pages of the book comprise a numerical index of all the patents mentioned, an index to the commercial products, and corrections to the preceding volume.

The five preceding volumes are considered by many to comprise a reference book of unsurpassed authority, a classic in its own field, and this additional volume certainly maintains that standard. In it Dr. Diserens gives a complete survey of the four textile processes mentioned above; but, although his approach is methodical and painstaking, it is also uncritical, and even unselective, especially in the lengthy paraphrasing of innumerable patents. Rarely does the author apply his critical faculties, and give the reader the benefit of his vast experience. Also, he has grouped his subjects so that there is some overlapping: e.g. methods for reducing the wet-swelling of rayon fibres are enumerated in the first chapter and again in the chapter on crease-resistance.

It is possibly a sign of the rapid progress made in resin finishing during the last few years that easy-care finishes on cotton are not mentioned. In fact, it is stated repeatedly (e.g. on pp. 337 and 385) that cotton cannot be crease-resisted satisfactorily. Such statements at once date the book, which, contrary to what the title suggests, is not so much about the latest developments as a survey of finishing methods, past and present.

In a book of this magnitude, it is not surprising that errors, printing and otherwise, have crept in. For example, Catalyst AC (Monsanto) is repeatedly stated to be triethanolamine hydrochloride, whereas in fact it is 2-amino-2-methylpropanol hydrochloride. Beetle textile resins are no longer available from Beetle Products Co., but from British Industrial Plastics Ltd.; and "polythene" is described as a du Pont trade-mark. Also the reader soon stumbles across errors due to insufficient checking, especially of names and patent numbers. Although these are minor points, which do not detract from the value of the book, they ought to be corrected in any future edition.

The printing and the paper are of a very high standard, in keeping with the traditions of this well known Swiss publishing house. This volume will undoubtedly find a place in the libraries of institutes, colleges, and universities concerned with research in textile chemistry, and will be even more valuable to progressive dyers and finishers. The price quoted, considering the compass of the work, does not appear to be unreasonable.

G. W. Madaras

Surface Active Agents and Detergents
Volume II

By A. M. Schwartz, J. W. Perry, and J. Berch. Pp. xv + 839. New York and London: Interscience Publishers. 1958. Price, $17.50.

The present volume is a volume of addition to *Surface Active Agents: Their Chemistry and Technology*, by Schwartz and Perry (cf. J.S.D.C., **65**, 454 (1949)), and is devoted to developments during the period 1947–1956. After an introductory survey, the book is divided into four main parts, dealing respectively with processes for synthesising and manufacturing surface-active agents, surface-active agents and compositions with special functions and properties, the physical and colloid chemistry of surface-active agents, and their practical applications.

This book can be warmly recommended to all workers in this field, and fully deserves the adjective "indispensable". The coverage of the

literature— scientific, technological, and patent— is very full. Although it is presumably written primarily for the American reader, the British and European literature appears to be very well covered.

In a comprehensive treatise of this sort, some portions will, of course, be of greater interest to some readers than to others. Among the sections of the book which seemed to your reviewer to be of particular interest or value were the summaries of surface-active agents for non-aqueous media, of the preparation of special physical forms of surface-active agents, of detergent processes, and of some of the more unusual applications of surface-active materials. Other readers will undoubtedly have other preferences, and the only really valid advice which can be given is that they should explore the book for themselves— and in not too great a hurry.

As will be apparent from what is said above, the book is largely a work of reference rather than a monograph, in the sense that its chief aim would appear to be completeness. The treatment is by no means uncritical, however, and the book is far more than just an impressive collection of facts.

Your reviewer hopes to be forgiven if, in spite of the excellence of this book, he nevertheless continues to use the phrase *surface-active agent* rather than the admittedly shorter "surfactant"!

J. L. Moilliet

Textile Chemicals and Auxiliaries

with special reference to Surfactants and Finishes

Edited by Henry C. Speel and E. W. K. Schwarz. 2nd edition 1957. Pp. vi + 545. New York: Reinhold Publishing Corpn. (London: Chapman & Hall Ltd.). Price, 108s. 0d.

Chemicals are assuming an increasingly important rôle in textile technology, and it is claimed that since 1953 the textile-producing and -processing industries have been consuming 25% or more of all industrial chemicals produced in the United States. It is necessary, therefore, that the industrial chemist should know something about textile fibres and the many techniques now adopted for converting them into finished cloth. It was to give a general account of the uses of chemicals in the textile industry that the first edition of *Textile Chemicals and Auxiliaries* was published. That it met a demand is evident from the issue of a second edition. The general lay-out of the book remains identical with that of the first edition. The work is divided into two parts: Part I is an account of the physical and chemical nature of fibres, the preparation of fibres and fabrics, dyeing and printing, application of pigments, textile finishing, fabric stabilisation, coated fabrics, and non-woven fabrics; while Part II is concerned with the textile uses of reagents such as water, oils, fats and waxes, starches, gums and glues, solvents, surfactants, soaps, synthetic resins and substances used in waterproofing, mothproofing, and flameproofing.

The present edition has been brought up to date by the inclusion of information about recent developments, e.g. bulked yarns, the production and colouring of the newer man-made fibres, the Avcoset process, flameproofing with trishydroxymethylphosphonium chloride, and mothproofing agents such as Dieldrin, Edolan FL, and Edolan WA. A new chapter on the important topic of felts and non-woven fabrics has been added, and some interesting data on the economic aspects of textile chemicals and auxiliaries have been collected together. Each chapter in the book has been written by a specialist in the field, but a surprisingly high degree of uniformity of presentation has been maintained throughout the work.

The task of writing a book covering such a wide range of subjects as "textile chemicals" is a formidable one, for so much material is available that the main difficulty is to decide what to omit. The authors have, however, made a wise selection, and have presented the information in a clear and systematic manner. A new style of type has been used for the new edition, and this makes the book much easier to read. Because so much ground is covered, the treatment of each topic is necessarily superficial, but the book as a whole will be extremely valuable to chemists interested in textiles and to textile men wishing to know something of the chemicals at present used in their industry.

C. S. Whewell

Chemistry of Carbon Compounds

A Modern Comprehensive Treatise

Volume IV Part A

Heterocyclic Compounds

Edited by E. H. Rodd. Pp. xxvi + 807. Amsterdam: Elsevier Publishing Co. (London: Cleaver-Hume Press Ltd.). 1957. Price, 147s. 0d.

The present volume, which introduces heterocyclic chemistry, follows a most successful series of six books covering aliphatic, alicyclic, and aromatic chemistry. The same competence in handling the material is to be seen as in the earlier volumes, and the generous use of formulae simplifies the reading throughout.

This book contains a systematic treatment of compounds containing three- to five-membered heterocyclic rings; pyrrole, furan, and thiophen together with their benzo compounds are comprehensively covered. There is, too, a major section on six-membered rings containing one nitrogen which deals with pyridine and its benzo compounds.

The presentation appears to be more condensed than in earlier volumes, with an increasing use of smaller type, doubtless necessary to keep the heterocyclic work in the three parts now suggested. Reference to the compression is made in the penicillin section, in which only "the barest outline of the extensive work . . . can be attempted here". Nevertheless, a worthwhile presentation adequately covers the field for all except the research specialist.

The useful references to original literature show how much compilation of recent work has been possible and reflects great credit on the six contributors to this part.

In a short introduction, the naming and the numbering of heterocyclic rings are briefly but

clearly presented. The index occupies about 10% of the pages and tends to be too complete; e.g. almost a dozen references are made to indigo, yet the colouring matter is to be dealt with in a later volume.

Reviews of the series so far have described each book as excellent. There must be few chemists who are not already convinced of the value of the volumes and await the completion of a superb work of reference for organic chemistry.

G. R. Ramage

Tracer Applications for the Study of Organic Reactions

By John G. Burr, Jr. Pp. x + 291. New York and London: Interscience Publishers. 1957. Price, $7.50 or 58s. 0d.

The increased availability of isotopic tracers has led to their wide application in many sciences in recent years. Essentially they provide "labelled" atoms which enable reaction systems to be relatively easily studied, and indeed many reactions can be followed in no other way than by their use.

The book under review is an account of tracer applications in the field of organic reaction mechanisms. Their value in this particular context is apparent from the number and the variety of reactions utilising them, which are most fully discussed in this book. The scope of the book is comprehensive, and the author's style is lucid. The approach is academic, and if the book is read in conjunction with standard texts on "mechanisms", it should give increased insight into this fascinating topic. It would be of particular value to teachers and research workers in this field. The treatment is somewhat too full for the general reader. The author has deliberately concentrated on the organic reaction chemistry, and no attempt has been made to give practical details of the preparation of isotopically labelled compounds or of tracer techniques. However, an appendix lists suitable texts, and throughout the book there are many references to the relevant research papers.

A few mistakes were noticed, e.g. the important exchange equations (6)–(9) on p. 16 all require correction, and it is interesting to note that, according to a reference on p. 247, isotopic tracers were in use in the year 1038! The style of print used by the publishers is not considered by the reviewer to be very satisfactory, and, as is usual with American books, the price is high.

G. J. Weston

The Chemistry of the Co-ordination Compounds

(American Chemical Society Monograph Series No. 131)

Edited by John C. Bailar, Jr., assisted by Daryle H. Busch. Pp. x + 834. New York: Reinhold Publishing Corpn. (London: Chapman & Hall Ltd.). 1956. Price, 148s. 0d.

This is a first-class book. Although the volume is published as an A.C.S. monograph, one gets the impression that each chapter is in itself a complete monograph on one particular aspect of co-ordination chemistry. Twenty-four authors have collaborated in writing this work, and the editor, Professor Bailar, is to be congratulated on his choice and arrangement of material, such that very little overlap occurs from one chapter to another. Professor Bailar states in his preface that "no attempt has been made to cover the chemistry of co-ordination compounds completely", and thus some topics, e.g. methods of preparation, have not been discussed at all. Nevertheless, a vast amount of information has been collected together, and the book is likely to be a standard reference for many years to come. It is not a book of practical "know-how" but rather a scholarly, yet readable, account of the theory and the applications of co-ordination chemistry.

Of the twenty-three chapters in the book, the last five are devoted to applied co-ordination chemistry. There are among these very useful accounts of co-ordination in dyes and pigments and co-ordination in analytical chemistry. The earlier chapters deal, in general, with theoretical aspects; thus the stereochemistry of these compounds is dealt with very fully. The nature of the co-ordinate bond is discussed both from the electrostatic and from the electron-pair points of view. Chapters on the poly-acids and the carbonyls and nitrosyls are among the many good things contained in the book. The editor contributes an interesting account of the early history of co-ordination theory up to and including Werner's epoch-making work.

In conclusion, this book is to be highly recommended to all whose interests touch upon co-ordination chemistry. It is unfortunate in a work of this kind that no author index is provided. However, well over 3000 literature references are given throughout the text. The book is delightfully produced and errors are extremely rare. The price puts the book beyond the reach of most individuals.

G. J. Weston

Fibre Microscopy

Its Technique and Application

By J. L. Stoves. Pp. vii + 286. London: National Trade Press Ltd. 1957. Price, 50s. 0d.

This book sets out to be a good many things: a history of the subject, an account of the instruments and their use, a description of the structure and appearance of fibres, a practical handbook for their identification and reactions. It is too much for one man and 286 pages. Inevitably, some aspects of the subject have to be dismissed too briefly.

An interesting, if chatty, introduction beginning with a brief historical survey, which seems to imply that history ended with Abbé, and which ranges from archaeology to criminology, makes one feel at the outset that the author could have spread himself more or, by attempting less, have tantalised less.

The first half describes the various instruments and how to use them. The treatment is clear and of immediate practical value, but there is not much theory. All forms of microscopy, including electron microscopy, are described, with some

indication of when to use the various methods and the sort of results which have been obtained with them.

However, the author is most at home with straightforward light microscopy, and one looks in vain for applications of the more recent methods and instruments in Part II of the book, which deals with the "Microscopy of Fibrous Materials". Here we find really practical advice on how to handle fibres for identification and research, using mainly the compound light microscope.

A more numerous and varied selection of micrographs, many perhaps of higher magnification, could have been included here with advantage. Although the book does not attempt to be an atlas of photomicrographs, inevitably one needs to refer to the photographs in order to understand the text, and further, when using the book as an aid to fibre identification and reactions, one needs really good micrographs.

The book is pleasant and easy to read. If you want to know a useful amount, but not too much, about microscopes and how to use them, it can be recommended. Textile microscopists will find it a useful introduction, a good practical handbook, and a valuable source book of references to works giving more detailed treatments, which they will undoubtedly find they will need. It is too expensive.

E. H. MERCER

Present Day Views on Mechanism of Leather Dyeing and their Practical Value

By G. H. Otto. Pp. 32. Ludwigshafen: Badische Anilin- & Soda-Fabrik A.G. [1956.] No price.

This booklet gives an account of a lecture delivered both in Leeds and in London in November 1955 by Dr. Otto, who is the outstanding research worker in leather dyeing.

Most of the material has been available already as a series of papers mainly in *Das Leder*. These were issued by the same firm in 1957 as a book *Beiträge zur Kenntnis der Wechselwirkungen im System Haut–Gerbstoff–Farbstoff* and part had earlier been published in an English translation as *Contributions to the Study of the Interactions in the System Hide–Tannin–Dyestuff*. Nevertheless, the new booklet is very useful in that it draws attention to a potentially important subject, and it can be described as making the first, though very tentative steps towards providing a chapter on leather dyeing for the next edition of "Vickerstaff".

Mainly as a result of Dr. Otto's advocacy, the basic ideas on the surface charge of collagen and how it is affected during leather manufacture are now generally accepted, but when it comes to calculating affinities for four acid dyes of increasing number of sulpho groups, the author seems to be misusing theory. The absence of the full experimental data makes a detailed appreciation impossible and, unfortunately, the booklet is characterised throughout by a quite un-Germanic lack of thoroughness. Thus the term "gross dissociation constant" is used, but not explained (see Elöd and Fröhlich, *Melliand Textilber.*, **22**, 622 (1951)) and the "pH limit" of Fig. 7 is defined on p. 10 as the pH where maximum uptake is reached, but on p. 27 as the pH to which the dye is soluble without aggregating. Such faults have arisen through hasty compilation. Though some dye formulae have become successively more acceptable, at least eight still remain notably unsatisfactory.

H. E. NURSTEN

New Books received

Notes on Work Study. No. 3—Case Histories from smaller firms and individual plants in the Chemical Industry. London: Association of British Chemical Mfrs. [1958]. Pp. v + 41. 4s. 0d.

Khimiya krasitelei. B. M. BOGOSLOVSKII and N. G. LAPTEV. Moscow: Gizlegprom. 1957. Pp. 435. 10 roubles 45 kopeks.

Yearbook 1958. Complete to 31st December 1957. London: British Standards Institution. [1958]. Pp. iv + 515. 15s. 0d.

Hydrogen Ions. Their Determination and Importance in Pure and Industrial Chemistry. H. T. S. BRITTON. Volume II. Fourth edition 1956. London: Chapman & Hall Ltd. Pp. xix + 489. 70s. 0d.

The Chemistry and Technology of Naphthalene Compounds. N. DONALDSON. London: Edward Arnold (Publishers) Ltd. 1958. Pp. xv + 512. 90s. 0d.

1958 Year Book. Philadelphia: Federation of Paint and Varnish Production Clubs. [1958]. Pp. 176. $2.50.

An Introduction to Electronic Absorption Spectroscopy in Organic Chemistry. A. E. GILLAM and E. S. STERN. Second edition 1957. London: Edward Arnold (Publishers) Ltd. Pp. xi + 326. 50s. 0d.

The Petroleum Chemicals Industry. R. F. GOLDSTEIN. Second edition 1958. London: E. & F. N. Spon Ltd. Pp. xvii + 458 + 6 folding tables. 95s. 0d.

The Reproduction of Colour. R. W. G. HUNT. London: Fountain Press. 1957. Pp. 208 + 13 art paper inserts. 63s. 0d.

Veredler-Jahrbuch— Deutscher Färberkalender für das Jahr 1958. **62** Jahrgang. O. MECHEELS (editor). Munich: Franz Eder Verlag. Pp. iv + 64 + i + 440 + 64 + 59. DM 13.00.

Progress Report of the Standing Technical Committee on Synthetic Detergents. London: Her Majesty's Stationery Office. 1958. Pp. 8. 6d.

Synthetic Detergents. A Study of the Development and Marketing of a New Product. P. A. R. PUPLETT. London: Sidgwick & Jackson Ltd. 1957. Pp. 241. 42s. 0d.

Proceedings of the Second International Congress of Surface Activity. J. H. SCHULMAN (editor). London: Butterworths Scientific Publications (New York: Academic Press Inc.). Volume **I**— *Gas–Liquid and Liquid–Liquid Interface.* Pp. xi + 521. 90s. 0d. Volume **II**— *Solid–Gas Interface.* Pp. viii + 348. 75s. 0d. Volume **III**— *Electrical Phenomena and Solid–Liquid Interface.* Pp. x + 621. 100s. 0d. Volume **IV**— *Solid–Liquid Interface (Washing, etc.) and Cell–Water Interface.* Pp. viii + 352. 75s. 0d. (£16 0s. 0d. or $50.00 for the set).

Die Physik der Hochpolymeren. Volume **IV**— *Theorie und Molekulare Deutung Technologischer Eigenschaften von Hochpolymeren Werkstoffen.* H. A. STUART (editor). Berlin: Springer-Verlag. 1956. Pp. xx + 667. DM 89.00.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

Pressure Dyeing and Washing Machine

S. N. Peshekhonov

Tekstil. prom., **18**, 33–34 (Jan. 1958)

An improved machine for the dyeing (with sulphur dyes) and washing of fabrics under pressure, consisting of two- and three-box dyeing and washing sections respectively, is claimed. G.J.K.

PATENTS

Apparatus for adding a liquid to a viscous spinning solution

Algemene Kunstzijde Unie *BP* 777,990

Improved mixing with less wear on the pumps is obtained by injecting the liquid into the axial portion of the stream of viscous spinning solution. W.G.C.

Scray

J. D. Robertson *BP* 789,776
BP 791,577

A scray in which materials of widely different kinds and characteristics may be accumulated without their becoming crushed or creased. C.O.C.

Sample Dyeing Machine

English Sewing Cotton Co. *BP* 791,597

An auxiliary container is placed inside a main container. The auxiliary container can be securely closed to seal its contents from liquid in the outer container. The sample and the necessary dye liquor are placed in the auxiliary container which then rotates and is heated by the liquid in the main container. C.O.C.

Pleated Introduction of Cloth into the Clip or Pin Chains of Tenters

H. Krantz Sohne *BP* 792,355

Solo-matic Bleaching System (VII p. 497)
Automatic Temperature Control in Silk Dyeing (VIII p. 498)

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Thickening Agent for Printing with Vat Dyes

N. P. Batsyn

Tekstil. prom., **17**, 35–36 (Aug. 1957)

Waste products from the production of alcohol can be used as a thickening agent in printing with vat dyes. The new agent, which is neutral to weakly acid, keeps well and compares favourably with starch. G.J.K.

PATENTS

Gas-fume Fading Inhibitors for Use in Spinning Solutions

Celanese Corpn. of America *BP* 792,337

The polymers of an ethylenically unsaturated compound containing a pyridine group, e.g. a polymer of 2-vinyl-5-methyl pyridine, are excellent gas-fume fading inhibitors for incorporation into solutions of cellulose acetate before they are extruded. C.O.C.

Reducing the Foaming Tendency and Enhancing the Wetting Power of Mercerising Lyes

Ciba *BP* 791,166

A mixture of an acid sulphate of 2-ethylbutanol-1 with either a sparingly water-soluble condensate of an alcohol of > 2 C and $\not< 1$ mol. each of ethylene and propylene oxides or an acetal of such an alcohol, and a solution promoter for the condensate or acetal is used, e.g. an aqueous solution of diethyleneglycolmonoethylether, 2-ethyl-hexanol-1, 1-(β-butoxy-ethoxy)-propanol-2, Na salt of the acid sulphate of 2-ethyl-hexanol-1 and the Na salt of the acid sulphate of 2-ethyl-butanol-1. C.O.C.

Water-repellent Agents

Bradford Dyers' Assocn. *BP* 791,117

Organosilicon compounds used in conjunction with compounds containing at least two *iso*cyanate or *iso*thiocyanate groups or their adducts are good water-repellent agents. Thus Terylene satin treated with a silicone and *m*-toluylene di*iso*cyanate has almost the same water-repellent efficiency as a similar fabric treated with the silicone in presence of butyl titanate, but the former finish is much more resistant to weathering. C.O.C.

Flame-resisting Agents for Cellulose

Gy *BP* 790,663

Water-soluble orthophosphoric acid derivatives in which at least one OH group of the H_3PO_4 is replaced by NH_2 or, preferably, NHAlk, and the other OH groups are replaced by aliphatic amine, alkoxy or alkyl groups, no organic radical having > 4 C, are used as flame-resisting agents in conjunction with methylol melamines, which may be etherified, as binding agents, e.g. a mixture of—

$$H_3C{\cdot}NH{\cdot}P\overset{\diagup NHCH_3}{\underset{\diagdown NHCH_3}{=O}} \quad \text{and trimethylol melamine.}$$

C.O.C.

Water-dispersible Solid Nitrilo-Methylolphosphorous Polymers—Flame-resisting Agents

Albright & Wilson Ltd. *BP* 790,641

Products which impart a flame-resisting finish to textiles are obtained by reacting suitable phosphorous and nitrogen compounds together in aqueous medium to a controlled extent, isolating the polymer formed and dehydrating it without causing further polymerisation. Thus, tetrakis (hydroxymethyl)phosphonium chloride (3 parts) was dissolved in water (10) and melamine (1·1) added. The mixture was heated almost to boiling, to the clear solution urea (3) was added and the whole boiled for 15 min. It was then cooled for 15 min. and poured into ethanol (400 by vol.). The grainy white precipitate was stirred well, filtered off, washed with 95% ethanol and air dried. It remained completely water-soluble even when kept for 3 weeks. C.O.C.

Flame-retarding Composition

Associated Lead Manufacturers *BP* 791,802

A flame retarding composition for cellulosic materials consists of a mixture of antimony trioxide (10–20%), plasticiser (30–50) and polyvinyl chloride or the like (to bring to 100). The plasticiser consists of a mixture of a plasticiser containing no chlorine and one containing chlorine, the total plasticiser containing $\not< 15\%$ by weight of chlorine, e.g. 36% polyvinyl chloride, 23% dioctyl phthalate, 23% Aroclor and 18% Sb_2O_3. The flame-retarding finish imparted resists repeated washing, scrubbing and dry-cleaning. The treated material has a soft handle.

BP 791,803

An aqueous dispersion of polyvinyl chloride or the like, Sb_2O_3 and a plasticiser is used, the dispersion being made by first dispersing the Sb_2O_3 in the plasticiser, emulsifying this dispersion in water and mixing the emulsion with a latex of polyvinyl chloride or the like. This dispersion is much more stable and gives a better flame retarding finish than if the Sb_2O_3 was dispersed in the aqueous phase of the emulsion. C.O.C.

Flame-resisting Finishing Composition

American Cyanamid Co. *USP* 2,784,159

A mixture of (1) a water-soluble salt of an oxygen-containing acid of pentavalent phosphorus, (2) a water-soluble polyalkylene polyamine, (3) cyanamide and/or lower alkylol cyanamides and/or dicyandiamide and/or NH_4 dicyandiamide and/or lower alkylol dicyandiamides and/or NH_4 cyanate and/or NH_4 thiocyanate and/or biuret and/or urea, and (4) a wetting and softening agent of the type described in *USP* 2,427,242 (J.S.D.C., **65**, 123 (1949)), is used for imparting a flame-resisting finish to textiles. It can be sprayed on as an aqueous solution and yields a soft handling fabric. An example is diammonium phosphate (100 parts by wt.), dicyandiamide (18·8), hexamethylene tetramine (6·4), softener (example 4 of *USP* 2,427,242) (6·3). C.O.C.

Clear and Pigmented Oil-in-Water Resin Emulsions
L. Auer *BP* 792,466

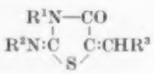
Emulsions suitable as binders for pigments for application to textiles are made from (*a*) a styrenated fatty-acid modified alkyd resin, a styrenated fatty oil or a pentaerythritol-glycerine mixed ester of a maleic anhydride-modified fatty acid, each being a polyhydric alcohol ester of a polyunsaturated monocarboxylic acid derived from a natural fat or oil, and soluble in hydrocarbons; (*b*) an emulsifying agent, e.g. soap, or a fatty alcohol sulphate; (*c*) a protective colloid, e.g. casein or methylcellulose. Their use gives faster curing, increased toughness and better ageing qualities of the prints. C.O.C.

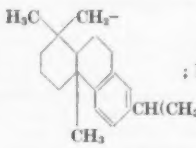
Tanning Agents
FBy *BP* 792,549

Cationic polyphenolic condensates of 1 mol. of a polyhydric phenol, $\not<$ 1 mol. of an amine or ammonia, and an aldehyde are excellent tanning agents despite their strongly basic character. C.O.C.

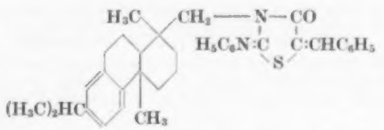
Thiazolidone Compounds as Ultraviolet Radiation Absorbants
Eastman Kodak Co. *USP* 2,784,087

Compounds of formula —



(R¹=  ; R² = Ar or aralkyl; R³ = Ar)

e.g.



are excellent absorbers of ultraviolet radiation. C.O.C.

Stabilised Solutions of Acrylonitrile Polymers
Chemstrand Corpn. *BP* 793,482

Addition of 0·01–2·0% of phenol, hydroquinone, 2:5-di-*tert*.-butylhydroquinone or 2:5-di-*tert*.-amylhydroquinone prevents solutions of acrylonitrile polymers in organic solvents discolouring when kept for a long time at high temperatures. C.O.C.

Coating Composition (V p. 496)
Plasticisers having Heat and Light Stabilising Properties for Vinyl Halide Polymer Compositions (XIII p. 503)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Basicities of *N*-Alkylanilines
H. T. Taylor
Nature, **181**, 265 (25 Jan. 1958)

Variation of pK_a values for several *N*-alkylanilines cannot be explained simply on the basis of steric inhibition of resonance in the free base, which would predict an increase with increasing size of alkyl group. Large alkyl groups introduce intramolecular overcrowding in the corresponding anilinium ions, leading to a fall in basicity. R.B.B.

Reduction of Nitrobenzene and its Derivatives
IX—Preparation of Hydrazobenzene using Iron as Reducing Agent
H. Iida
Rept. Govt. Chem. Ind. Research Inst. Tokyo, **52**, 419–422 (lii) (Dec. 1957)

In the preparation of hydrazobenzene by the reduction of nitrobenzene in caustic soda solution with ferrosilicon as reducing agent, the addition of lime to the reaction mixture makes the reduction proceed smoothly.

X—Applicability of Ferrosilicon to Organic Reductions
H. Iida and K. Kayahara
Ibid., 423–426 (liii)

Organic compounds have been reduced with ferrosilicon in caustic soda solution in order to study what kinds of organic compounds are reduced by this process. The results obtained are as follows—(1) In the reduction of nitrobenzene derivatives, hydrazo compounds are obtained in the same good yield as in reduction with zinc dust. (2) Phenolphthalein, anthraquinone, and benzyl chloride are reduced to phenolphthalin, anthraquinol and toluene respectively. The groups $>$CO and –CH:N–, however, are not reduced to $-CH_2-$ and $-CH_2\cdot NH-$ groups respectively, because the reducing power of ferrosilicon is weaker than that of zinc dust activated with copper. C.J.W.H.

Thermal Decomposition of 3:5-Dibromobenzene-1:4-diazo-oxide
M. J. S. Dewar and A. N. James
J.C.S., 917–922 (March 1958)

3:5-Dibromobenzene-1:4-diazo-oxide decomposes in chlorobenzene above 70°C. to give nitrogen, bromine and thermally stable polymers, which appear to be copolymers of bromochlorodiphenylyloxy and dibromophenoxy units. Similar results are obtained on the thermal decomp. of 3:5-dichlorobenzene-1:4-diazo-oxide in chlorobenzene. These 3:5 deriv. were employed to avoid side-reactions at the reactive positions *ortho* to the oxygen in the diazo-oxide. The salient features of the reactions are the evolution of free halogen and the incorporation of solvent mol. into the polymer, indicating free-radical intermediates and radical substitutions. H.H.H.

Reaction Mechanism of the Formation of Azoxybenzene
M. M. Shemyakin, V. I. Maimind, and B. K. Vaichunaite
Izvestiya Akad. Nauk S.S.S.R., otdel khim. nauk, 1260–1262 (Oct. 1957)

Azoxybenzene is prepared from phenylhydroxylamine and nitrosobenzene (^{15}N) and the reaction mechanism studied by mass-spectrometry after bromination of azoxybenzene and subsequent reduction to aniline and *p*-bromoaniline. The authors postulate the following mechanism—

$$C_6H_5{}^{15}NO + C_6H_5{}^{14}NHOH \rightarrow C_6H_5\cdot{}^{15}\underset{\displaystyle OH}{N}\text{——}{}^{14}\underset{\displaystyle OH}{N}\cdot C_6H_5$$

$$\xrightarrow{-H_2O}\left[\tfrac{1}{2}\,C_6H_5{}^{15}\underset{\displaystyle \overset{\downarrow}{O}}{N}{:}{}^{14}N\cdot C_6H_5 + \tfrac{1}{2}\,C_6H_5\cdot{}^{15}N{:}{}^{14}\underset{\displaystyle \overset{\downarrow}{O}}{N}\cdot C_6H_5\right]$$

since the labelled nitrogen is equally divided between the two reduction products. G.J.K.

Phenylation with Diazoaminobenzenes
R. L. Hardie and R. H. Thomson
J.C.S., 1286–1290 (March 1958)

Pyrolysis of diazoaminobenzenes in 4% soln. of the aromatic solvents bromo-, chloro-, nitro-, and *iso*propylbenzenes at 150–160°C. for 40 hr. generates phenyl and anilino radicals; the former attack the solvent, giving relatively high yields (usually $>$ 50%) of diphenyl deriv. and negligible amounts of by-products. Thus the pyrolysis of the diazoaminobenzenes affords yet another method of phenylation, which has the advantage over other procedures that the reactions are clean. Three out of four unsymmetrical diazoaminobenzenes examined gave mixtures of amines and diphenyls which show that these triazens undergo decomp. in both tautomeric forms; however, 4-bromodiazoaminobenzene reacts exclusively in one tautomeric form in boiling bromobenzene. H.H.H.

New Direct and Vat Dyes
P. A. Simigin and M. Z. Tangiyan
Tekstil. prom., **17**, 32–34 (Aug. 1957)

Nine vat dyes and 10 direct dyes are tabulated, and their fastness to light, weathering, water, washing (at 40° and 100°C.), perspiration, ironing, dry and wet rubbing, bleaching, and fading on rubbing are given. G.J.K.

Relation Between the Absorption Spectra and the Chemical Constitution of Dyes. XXIX—Interaction of Direct Azo Dyes in Aqueous Solution
M. N. Inscoe, J. H. Gould, M. E. Corning, and W. R. Brode
Bur. Stand. J. Res., **60**, 65–83 (Jan. 1958): *Research Paper* 2823

Mixtures of direct azo dyes generally give non-additive spectra, indicating interaction between the dyes in soln.

Acid azo dyes show less evidence of interaction, but may interact with direct azo dyes. Interaction is lessened by additions of ethanol or non-ionic detergents, and increased by inorganic salts. The tendencies of direct dyes to interact generally follow the tendencies of the individual dyes to aggregate and to show substantivity for cellulose. It is suggested that the same forces are operating in each case. A.J.

Derivatives of *m*-Trifluoromethylaniline and Fluorine-substituted Naphthol AS (C.I. Azoic Coupling Component 2)
K. Inukai and K. Hosokawa
Kôgyô Kagaku Zasshi, **59**, 441–443 (1956): *Chem. Abs.*, **52**, 2412 (10 Feb. 1958)

m-$CF_3 \cdot C_6H_4 \cdot NH_2$ substituted with Cl, $SO_2 \cdot C_2H_5$ and $O \cdot CH_3$ in *ortho*-positions when coupled with Naphthol AS and F-substituted Naphthol AS yielded orange–yellow to red dyes of good fastness on cotton. The absorption spectra and m.p. of the dyes are given. C.O.C.

Synthesis of Surface-active Dyes and their Properties
V. O. Yoda
Nippon Kagaku Zasshi, **78**, 631–635 (1957): *Chem. Abs.*, **52**, 2411 (10 Feb. 1958)

The critical micelle concentration of the surface-active dye octylaniline → naphthionic acid cannot be determined by means of the fluorescence of Rhodamine 6G (C.I. Basic Red 1). The intensity of the fluorescence of the mixture of this dye + Rhodamine 6G + dodecylpyridinium bromide changed when the proportions of the components were altered. C.O.C.

Thianthren Azo Dyes for Wool
A. Cerniàni
Boll. sci. Fac. chim. ind. Bologna, **15** (3), 98–99 (1957): *Chem. Abs.*, **52**, 2411 (10 Feb. 1958)

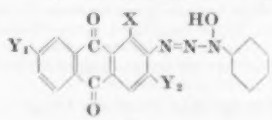
2-Aminothianthren was diazotised and coupled in alkaline medium with chromotropic acid and the dye salted out at 5°c. with 20% NaCl and then purified via the Ba salt. Similar dyes were made using H and R acids and from 2:7-diaminothianthren and chromotropic acid. The dyes were applied to wool using ammonium acetate as the assistant and exhausting by adding Na_2SO_4 and acetic acid. C.O.C.

Lattice Image of Twin Structure observed directly by Electron Microscope in a Crystal of Copper Phthalocyanine
E. Suito, N. Uyeda, H. Watanabe, and T. Komoda
Nature, **181**, 332–333 (1 Feb. 1958)

High-resolution electron microscopy with the stable form of copper phthalocyanine has shown the existence of two kinds of crystal habit in single crystals. The two habits are discussed. A.J.

Hydroxydiazoamino Compounds of Anthraquinone
V. Ya. Pochinok, V. P. Pompa, and O. D. Tonkoshkur
Ukrainian Chem. J., **23**, 629–633 (Oct. 1957)

The synthesis of six new hydroxytriazens of anthraquinone derivatives (e.g. I) from diazotised α- or β-aminoanthraquinone and phenylhydroxylamine is given.



(I)

(X = NO_2 or Cl, Y_1 and Y_2 = H; or X = H, and Y_1 or Y_2 = SO_3Na). These compounds are of use as analytical reagents. G.J.K.

Meerwein Arylation of Anthracene, 9-Phenylanthracene, and 9-Anthroic Acid
S. C. Dickerman, L. B. Levy, and A. W. Schwartz
Chem. and Ind., 360 (22 March 1958)

Recent work by Dickerman and Weiss (*J. Org. Chem.*, **22**, 1070 (1957)) on homolytic aromatic arylation is now extended to the title substances. Anthracene is both 9-mono- and 9:10-diarylated with *p*-nitro-, *p*-chloro-, *p*-methoxy-, and benzenediazonium chlorides (I). The monoarylated deriv. have not been detected previously in such systems, and the positive results with I are in disagreement with the earlier work of Étienne and Dégent (*Compt. rend., Acad. Sci., Paris*, **236**, 92 (1953); **238**, 2093 (1954)). 9-Phenylanthracene is arylated in the 10-position with diazotised aniline and *p*-nitroaniline, and similarly, 9-anthroic acid gives 10-aryl-9-anthroic acids. The yields for these reactions are held to be in agreement with the concept of an intermediate of the triarylmethyl radical type. H.H.H.

Anthraquinone Dyes
A. Tundo
Boll. sci. Fac. Chim. ind. Bologna, **15** (3), 100 (1957): *Chem. Abs.*, **52**, 2411 (10 Feb. 1958)

1-Amino-2-sulpho-4-phenoxyanthraquinones having such substituents in the benzene ring as *o*-, *m*-, or p-CH_3, *o*- or p-$O \cdot CH_3$, or *o*- or *p*-Cl are orange to reddish orange. 1-Amino-2-sulpho-4-phenylthioanthraquinones having in the benzene ring such substituents as p-CH_3, *o*- or p-$O \cdot CH_3$, *o*- or *p*-Cl, or p-$O \cdot C_2H_5$ are red to violet; they are faster to light and washing than are the 1-amino-2-sulpho-4-phenoxyanthraquinones. Both series of dyes yield faster dyeings on cellulose acetate than on wool. C.O.C.

Constitution of Cernoside, the Yellow Pigment of the Flowers of *Oxalis cernua*
A. Ballio and G. B. Marini-Bettolo
Gazz. chim. ital., **85**, 1319–1328 (1955): *Chem. Abs.*, **52**, 1990 (10 Feb. 1958)

Cernoside is shown to be the 4-glucoside of aureusidine (3′:4:4′:6-tetrahydroxybenzylidenecoumaranone). C.O.C.

Nature of Homovitexin
W. B. Whalley
Chem. and Ind., 361–362 (22 March 1958)

The yellow pigment vitexin (I), obtained by A. G. Perkin from *Vitex littoralis*, was claimed by him to be closely associated with a second pigment, homovitexin (II). However, the recent elucidation of the relationship between I and saponaretin (which was thought to be identical with II), together with the failure of subsequent investigators to obtain II, has made the present definition of the status of II opportune. It now appears that when the very sparingly sol. I is dissolved in a large volume of alcohol, which is then gradually removed, I does not separate but that, when most of the solvent has gone, a microcryst. substance (III) appears. III does not depress the m.p. of I, has the same mol. rotation and infrared spectrum, and recrystallises from aq. pyridine to regenerate I. This result indicates the probable identity of III with II, from which it would follow that II is an amorphous variety of I. Unfortunately, Perkin's original specimen of II no longer exists for comparison purposes. H.H.H.

Production and Properties of Zinc Oxide (C.I. Pigment White 4)
G. Scacciati and P. Gondi
Chim. e Ind. (Milan), **39**, 832–841 (1957): *Chem. Abs.*, **52**, 3359 (25 Feb. 1958)

Description with illustrations of a pilot plant for the production of ZnO and an account of the pigment obtained, illustrated by photomicrographs showing the particle size. C.O.C.

PATENTS

1-Benzoylamino-4-*p*-benzene-azo-anilino-2-methylanthraquinone
ICI *BP* 792,717

This dye intermediate (cf. *BP* 560,988; J.S.D.C., **60**, 250 (1944)) is prepared by reacting 4-amino-1-benzoylamino-2-methylanthraquinone in a high-boiling organic solvent with a 4-halogenoazobenzene. An acid-binding agent and a catalyst, e.g. Cu powder, Cu acetate or Cu_2Cl_2 may be used. Thus, 4-amino-1-benzoylamino-2-methylanthraquinone (13), *p*-bromoazobenzene (13), anhydr. Na_2CO_3 (4·6), Cu bronze (0·3) and Cu_2Cl_2 (0·3) are heated in nitrobenzene at b.p. for 12 hr. The mixture is cooled, filtered, the residue being then washed with alcohol and boiled with dil. HCl to remove Cu salts. This process avoids use of 4-aminoazobenzene which is suspected of possessing carcinogenic properties and also gives better yields than the earlier method. E.T.

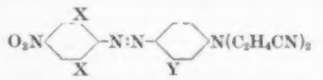
all-*trans*-Carotenoids
F. Hoffmann–La Roche & Co. *BP* 793,236

all-*trans*-C_{40}-carotenoids are obtained by heating suspensions of 15,15′-mono-*cis* C_{40}-carotenoids in an inert organic liquid. Thus all-*trans* β-carotene is obtained by heating 15,15′-monocis β-carotene in petroleum ether for 10 hr. at 80°c. in a CO_2 atmosphere. C.O.C.

Orange and Red Monoazo Disperse Dyes from *NN*-Bis(cyanoethyl)anilines

DuP *USP* 2,782,187

Monoazo disperse dyes—



(X = H or Hal; Y = H or Alk of 1–4 C) give oranges and reds of good fastness to light and sublimation on polyester fibres. Thus the monoazo dye *o*-chloro-*p*-nitroaniline → *NN*-bis(cyanoethyl)-*m*-toluidine is a red. E.S.

Orange and Brown Metal (Chromium and Cobalt)-complex Monoazo Pyrazolone Dyes for Wool, Nylon, etc.

ICI *BP* 792,206

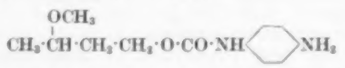
The 1:2 chromium(and cobalt)-complexes of the monoazo compound (I) 2-amino-4-nitrophenol → 3-methyl-1-*o*-toluyl-5-pyrazolone are more soluble than the homologues from 3-methyl-1-phenyl-5-pyrazolone, and dye wool and nylon orange to brown from neutral baths. Mixed complexes containing 1 atom of Co or Cr, 1 mol. of I, and 1 mol. of other suitable *oo'*-dihydroxymonoazo compounds free from SO_3H, COOH, SO_2NH_2 and sulphone groups, are also claimed. E.S.

Yellow Monoazo Disperse Dyes having a Carbamic Ester Group

S *BP* 792,212

Yellow monoazo dyes for cellulose acetate, nylon, polyester, and polyacrilonitrile fibres—

(R^1 = Alk, Ar, cycloalkyl, or aralkyl; R^2·OH = radical of a phenol which may carry substituents, especially acylamino; A may carry substituents such as Hal, Alk, NO_2, SO_2Alk) are made by coupling a diazotised 1-aminophenyl-4-carbamic acid ester with a suitable phenol, or by condensing a suitable *p*-aminomonoazo compound with a chloroformic ester. Thus *p*-aminoacetanilide is stirred at 0°C. with 3-methoxybutyl chloroformate in presence of aq. sodium acetate. Hydrolysis of the acetylamino group then gives the aminophenylcarbamic ester—

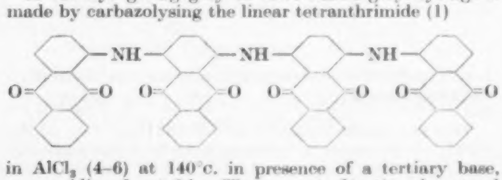


which is diazotised and coupled with *N*-acetyl-*m*-aminophenol to give a greenish-yellow disperse dye. E.S.

Anthraquinone Vat Dye

Ciba *BP* 792,036

A vat dye giving grey to olive-tinted grey dyeings is made by carbazolysing the linear tetranthrimide (I)



in $AlCl_3$ (4–6) at 140°C. in presence of a tertiary base, e.g. pyridine, for ≮ 3 hr. The amount of tertiary base used is such as to give an easily stirrable melt at 100°C., e.g. $AlCl_3$:pyridine : : 1 : 1·6 to 2. The product obtained by this method compares favourably with that given by the method of *BP* 693,075 (J.S.D.C., **69**, 386 (1953)) the latter being a reddish-grey which, although also having good light and washing fastness, is not fast to chlorite. E.T.

Alkylation Process for Dibenzanthrone Vat Dyes

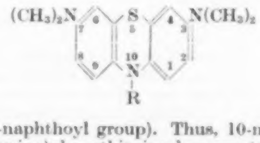
CCC *BP* 792,795

Complete alkylation of dihydroxybenzanthrones is achieved by (1) converting the dihydroxybenzanthrone to the salt of a volatile amine, (2) removing the water from the reaction mixture, (3) converting the amine salt to the alkali metal salt, and (4) removing the amine by distillation before finally alkylating. The reactions are carried out in an organic solvent, e.g. trichlorobenzene, nitrobenzene, and a slight excess of amine, preferably triethylamine, is used. The dihydroxybenzanthrone is used in the form of wet filter cake the water being removable as an azeotrope in stage (2) above; a maximum temperature of 160°C. is generally sufficient to remove the amine in stage (4). Alkylation proceeds at 135–200°C. using alkyl sulphates, alkyl aryl sulphonates, alkyl halides, etc. The alkylated product is separated by cooling and filtering and excess solvent is removed by steam distillation. The present method avoids the disadvantages of the usual method (*a*) by preventing oxidation of the dihydroxybenzanthrone to diquinone, (*b*) by thus eliminating the necessity for a later purification stage, and (*c*) by producing the alkali metal salt in a well dispersed form suitable for easy and complete alkylation. E.T.

Naphthoyl *leuco*-Methylene Blue—Transfer Coatings for Manifold Copy Sheets

National Cash Register Co. *USP* 2,783,227

Light-stable *leuco*-methylene blue derivatives are of structure—

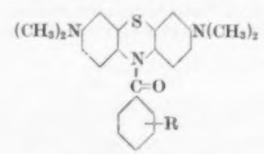


(R = 1- or 2-naphthoyl group). Thus, 10-naphthoyl-3:7-bis(dimethylamino)phenothiazine has greater stability to light in the form of its colourless *leuco*-compound than the corresponding benzoyl-derivative which decomposes to an undesirable blue-green colour. The naphthoyl derivative is prepared by adding 2-naphthoyl chloride (25) to the $ZnCl_2$ double salt of *leuco*-methylene blue (35) in pyridine (97) and refluxing for 1 hr. Pouring into cold water (1000) and washing by decantation with NaOH (17·5) in water (500) and then water alone follows. The semi-solid residue is extracted with benzene, treated with fuller's earth, filtered and the product is then precipitated from the filtrate by addition of petroleum ether. E.T.

Nitro-derivatives of Benzoyl *leuco*-Methylene Blue Transfer Coatings for Manifold Copy Sheets

National Cash Register Co. *USP* 2,783,228

The light-stable *leuco*-methylene blue—

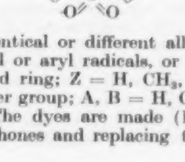


(R = 3- or 4-NO_2) is prepared, e.g. by reacting *p*-nitrobenzoyl chloride with *leuco*-methylene blue/$ZnCl_2$ double salt in pyridine under reflux. The products, and those from the method of *USP* 2,783,227 (see above), are used mixed with crystal violet lactone (3:3-*bis*(*p*-dimethylaminophenyl)-6-dimethylamino-phthalide) in oil and wax to give colourless to pale-cream coatings on manifold copy-sheets. The crystal violet lactone gives an immediate blue coloured impression on paper sensitised by impregnation with an acid clay but this fades relatively quickly and the copy is preserved by the slower developing blue produced from the *leuco*-methylene blue derivative. E.T.

Yellow to Orange-Red Disperse Dyes

BASF *BP* 792,396

Fast clear yellows to orange-reds are obtained by dyeing cellulose acetate, polyacrylonitrile, polyamide, polyurethane or polyester fibres with aq. dispersions of—



(R^1, R^2 = H, identical or different alkyl, hydroxyalkyl, *cyclo*alkyl, aralkyl or aryl radicals, or are members of a common saturated ring; Z = H, CH_3, C_2H_5, acyl, carboxylic acid or ester group; A, B = H, CH_3, C_2H_5, alkoxy, NO_2 or Hal). The dyes are made (1) by oxidising 1-halogenothioxanthones and replacing the Hal by amino

(2) by nitrating 2-alkylbenzophenone-sulphones, converting the product to its isoxazole derivative and then reducing in acid medium to give 1-amino-2-carboxy derivatives. If the fibre to be dyed has been pretreated with "animalizing" agents, e.g. the reaction product of acrylonitrile (2 mol.) and *pp'*-diamino-di*cyclo*hexylmethane, 1-aminobenzophenone sulphones containing COOH are best. E.T.

Chlorinated Cobalt Phthalocyanines
DuP *USP* 2,782,208

Intermediate complex compounds capable of producing polychloro-Co-phthalocyanines on reduction are of formula

$$C_{40}Cl_xH_{20-x}N_{11}Co$$

(x = 10–20). They may be made by reacting tetrachlorophthalic anhydride, or mixtures of tetrachlorophthalic anhydride and unsubstituted phthalic anhydride, with urea and cobaltous chloride using NH_4 molybdate as catalyst in trichlorobenzene. Reaction occurs at 140–180°c. giving a small amount of by-product Co phthalocyanine pigment and the desired complex compound which is not a pigment. The complex also differs from the pigment in being soluble in alcoholic solvents, e.g. lower monoalkylethers of ethylene glycol. Separation of the product from the melt is effected by (1) steam distillation and filtration followed by extraction of the complex with dimethylformamide and reprecipitation with water, or (2) filtration of the hot melt to remove the pigment and steam distillation of the filtrate to give the insoluble complex or (3) filtration of the hot melt followed by dilution of the filtrate with an organic solvent, e.g. *cyclo*hexane. These complexes may be applied, in solution, to textile fibres by dyeing or printing procedures and subjected to reduction with e.g. Na hydrosulphite in aqueous, alcoholic or aq. alcoholic medium to generate the green pigment. The dyeings have excellent light fastness and good washing fastness. E.T.

Copper Phthalocyanine (C.I. 74160)
DuP *USP* 2,782,207

An alcohol-soluble complex compound capable of giving Cu phthalocyanine by treatment with reducing agents is of formula—

$$Cu(C_8H_4N_2)_6NH$$

and is described as "Cu phthalocyanine precursor". Earlier patents, viz. *USP* 2,772,283 and 2,772,284, describe its production from phthalonitrile, anhydr. Cu salt and NH_3 in an alcoholic solvent with e.g. urea guanidine, methyl glucamine, etc. as catalyst at $\not>$ 90°c. and show that reaction time may be shortened by passing O_2, air or air/NH_3 mixtures through the melt. The present process uses a solid or liquid oxidising agent in the melt, e.g. alkali metal nitrites, nitrates, ferricyanides, nitrobenzene sulphonates, H_2O_2, HgO, etc. Thus, anhydrous $CuCl_2$ (22·5) is added to dimethylformamide (188) and the mass is saturated with NH_3 gas. Phthalonitrile (128) and methylglucamine (10) are added and the mass is heated at 81°c. Aq. H_2O_2 30% (10) in dimethylformamide (25) is added dropwise over 2 hr. at 81–84°c. The reaction mass is filtered to clarify and the product is precipitated by pouring into water. Filtration, water-washing, and drying follow. E.T.

S-Containing Phthalocyanine Dyes
CFM *BP* 792,018

Greenish-greys of outstanding light fastness are produced by dyeing vegetable fibres from an Na_2S bath with S-containing phthalocyanine dyes. The latter are made by reacting polyaminophthalocyanines (from nitrated phthalic acid) with $ClSO_3H$ at 100–150°c. and then reducing with Fe or Zn in mineral acid suspension. Thus, tetramino-Cu-phthalocyanine (12·7) and $ClSO_3H$ (190·5) are stirred 2 hr. at 125°c. The melt is poured on to a mixture of ice (857), conc. HCl (200) and Fe powder (40) and the temperature is raised to 85–90°c. within 24 hr. E.T.

Cyanine Dyes containing a *N*-carbamylmethyl Group
Kodak *BP* 792,380

Dyes of formula—

$$\overbrace{N\cdot(CH{:}CH)_n\cdot C}^{Z^1}{:}CH(CR^2{:}CH)_d\cdot \overbrace{C({:}CH\cdot CH)_q{:}N}^{Z^2}R^1X$$
$$\underset{CH_2\cdot CONR^3R^4}{|}$$

(R^1 = Alk; R^2, R^3 and R^4 = H or Alk; X = acid radical; d, n and q each = 0 or 1; Z^1 and Z^2 = atoms to complete 5-membered heterocyclic nuclei), e.g. 3-diethylcarbamylmethyl-2-methylbenzothiazolium chloride, have good photosensitising properties. C.O.C.

Carbon Black (C.I. Pigment Black 7)
Columbian Carbon Co. *USP* 2,782,101

Modification of *USP* 2,659,663, by producing a non-oxidising blast flame the yield of furnace black is considerably increased. C.O.C.

Furnace Black (C.I. Pigment Black 7)
Phillips Petroleum Co. *USP* 2,781,246

Process for producing a high structure pigment of great tinting strength.

USP 2,781,247

Plant for production of high quality black of fine particle size, high tinting strength and high surface area by pyrolysis and/or partial combustion of carbonaceous material.

USP 2,781,251

An improved type of quench for a carbon black furnace.

USP 2,781,250

An improved burner for carbon black furnaces burning vaporous fuel. C.O.C.

Alumina-coated Pigments
DuP *USP* 2,780,558

Pigments are dry ground with a little aluminium alkoxide to improve their colour retention properties. TiO_2 (C.I. Pigment White 6) treated in this manner used in synthetic resin stoving enamels retains its colour when baked at high temperatures. C.O.C.

Caramel (C.I. Natural Brown 10)
Union Starch & Refining Co. *USP* 2,784,118

Caramel of a rich brown colour and having foaming properties is obtained by heating the raw material with an alkaline reagent before carrying out the main caramellisation. C.O.C.

Fluorescent Pigment Compositions
Switzer Brothers *BP* 792,616

A fluorescent dye is incorporated into the co-condensate of (1) an aromatic sulphonamide containing > 1 reactive H atom, (2) urea, thiourea, dicyandiamide, guanidine, malonamide, biuret or succinamide and, (3) formaldehyde. The amine-formaldehyde component forms $\not>$ 50% by wt. of the co-condensate. Thus a sulphonamide–HCHO resin (17) is melted at 120°c; paraformaldehyde (13) and urea (10) are added and the mixture heated to 130°c. Rhodamine B (C.I. Basic Violet 10) and Rhodamine 6G (C.I. Basic Red 1) are added and the product ground on a hammer mill to yield a pigment for use in silkscreen printing, paints, inks, etc. C.O.C.

Henna Leaves (C.I. Natural Orange 6) for Dyeing Textiles (VIII p. 498)
10-(Halo-benzoyl)-3:7-bis(dimethylamino)phenothiazines for Use in Copy Printing (IX p. 500)
Rendering Pigments, Fibres and Other Fine Particles capable of being readily compounded with Organic Substances (XIII p. 503)
Identification of Pigments by Back Scattering of β-Rays (XIV p. 504)

V—PAINTS; ENAMELS; INKS

Extender Pigments in Blister-resistant House Paints
Northwestern Paint & Varnish Production Club

Offic. Dig., Federation Paint & Varnish Production Clubs, **28**, 1078–1082 (1956):
Chem. Abs., **51**, 17194 (20 Nov. 1957)

The extenders tested were $CaCO_3$ (ppt.), $CaCO_3$ (coarse, natural dry ground) (I) (C.I. Pigment White 18), Mg silicate (coarse medium consistency) (C.I. Pigment White 26), diatomaceous SiO_2 (C.I. Pigment White 27), Al silicate, mica (325 mesh) (C.I. Pigment White 20), barytes (C.I. Pigment White 22), Ca silicate, diatomaceous calcite (II), and $CaSO_4$ (in Ti–Ca pigment). The pigmentation consists of rutile (C.I. Pigment White 6) and the selected extender. The pigment volume concentrations (PVC) are 20, 30 and 40. The ratio of TiO_2 to non-volatile vehicle is held constant, and the amount of extender pigment is varied to produce the desired PVC. The vehicle is a blend of alkyd resin, conforming to *U.S. Federal Specification*

TT–R–266, Type 1, and a linseed copolymer resin. A paint conforming to *U.S. Federal Specification* TT–P–102, Class B, is used as the control. The coated wooden panels are aged in a Weatherometer for 100 hr. and then placed in a blister test box for 3 days. The box is the type developed by the Archer-Daniels-Midland Co. None of the extenders affected the blister resistance adversely. All of the 40% PVC paints, except I and II, tend to crack under these conditions. A blend of extender pigments is necessary to produce optimum properties. C.O.C.

PATENTS

Resin-bonded Pigment Dispersions
Rohm & Haas Co. *BP* 791,767

A film-forming pigmented composition is dispersed in an aqueous medium at pH 8·0–9·8 with the aid as emulsifying agent of a non-ionic interpolymer containing (*a*) units having carboxylate groups derived from a polymerisable carboxylic acid having α,β-unsaturation in a vinylidene group, (*b*) units from a neutral, free radical-polymerisable ester having a vinylidene group attached to the functional group, which ester by itself yields a soft linear homopolymer, and (*c*) units from a neutral polymerisable monovinylidene ester which by itself yields a hard homopolymer. Thus Carbon Black (C.I. Pigment Black 7) is ground with partially desulphonated sodium lignin sulphonate and then mixed with an aqueous dispersion of the ammonium salt of an interpolymer of methacrylic acid, ethylacrylate, and methylmethacrylate containing octylphenoxypolyethoxyethanol as the emulsifying agent. This yields a printing ink which yields a dense black with a semi-gloss. It sets within 5 sec. and dries within 5 min. to yield prints of high resistance to water. Printing pastes for use on textiles can be similarly produced. C.O.C.

Coating Composition
Sherwin-Williams Co. *USP* 2,780,555

A composition applicable in paints, adhesives, paper sizes, etc. comprises an ungelled aqueous solution of zirconyl ammonium carbonate and a metal ammonium alginate (the metal being either Cr, Mn, Fe, Co, Ni, Cu, Zn or Al). It may be used alone or as a binder for pigments. The films it yields have high resistance to alkali and washing. C.O.C.

Stabiliser for Paints containing Non-reactive Pigments
J. J. Taylor *USP* 2,783,159
USP 2,783,160

Addition of a little dibasic lead phosphite $2PbO \cdot PbHPO_3 \cdot \frac{1}{2}H_2O$, made by the process described in *USP* 2,483,469, to paints containing non-reactive (non-chalking) pigments, e.g. TiO_2 (C.I. Pigment White 6) talc (C.I. Pigment White 26), Al silicate and mica (C.I. Pigment White 20), stabilises them against scaling when exposed to sunlight and air. C.O.C.

Brushing Polychrome Finishes
Sherwin-Williams Co. *USP* 2,780,559

Coatings appearing to have different colours or depth of colour when viewed at different angles are obtained by use of a composition containing an organic film-forming binder in which is dispersed 0·075–0·3% (on wt. of the whole composition) of a non-drying alkyl silicone polymer of viscosity 10–120,000 centistokes and 5·5–13·5% of a non-leafing aluminium pigment (C.I. Pigment Metal 1) of particle size $<$ 100 mesh. If desired 15–50% on the weight of the pigment of a hydrophilic, siliceous flatting agent of particle size 50–200 mμ may be added to reduce the gloss; exceeding this amount of flatting agent results in no deep tone of colour being apparent. C.O.C.

Fluorescent Pigment Compositions (IV p. 495)

VI—FIBRES; YARNS; FABRICS

Textile Research Achievements in 1957
J.B. Goldberg

Canadian Textile J., **75**, 61–65 (7 Feb.), 64–71 (21 Feb. 1958)

Activation Analysis of Fibrous Materials
O. Teszler and H . A. Rutherford

Text. Research J., **28**, 86 (Jan. 1958)

Many fibres become radioactive when subjected to a high flux of neutrons, thus allowing trace elements (generally impurities) to be identified by appropriate nuclear techniques. ^{32}P has been identified in cotton and rayon, while ^{203}Hg, ^{140}Ba, and ^{126}I are suspected in secondary cellulose acetate. S.B.D.

Structure of Hydrated Cellulose Fibres
Z. G. Serebryakova and N. V. Mikhailov

Tekstil. prom., **17**, 19–22 (Sept. 1957)

The mechanical and physical properties of cellulosic fibres are correlated with the degree of hydration of natural and regenerated cellulose. The concept of a relative density coefficient of molecular packing is introduced. G.J.K.

Cortical Differentiation in Burmese Wool
W. R. Lang

Text. Research J., **28**, 90–91 (Jan. 1958)

The mean fineness and staple length of several samples are recorded. The finer fibres showed some cortical differentiation. As the fibre thickness increased, a central orthocortex became more common, and in the coarsest fibres there was no indication of any cortical differentiation. This is contrasted with results for Pakistani carpet wool. S.B.D.

Sorption Kinetics of Water Vapour in Wool Fibres
J. G. Downes and B. H. Mackay

J. Polymer Sci., **28**, 45–67 (Feb. 1958)

Studies are made of the sorption kinetics of water vapour in single wool fibres. Small changes in water content occur in two stages, one occupying a few minutes and the other many hours at room temperature. The first stage is considered to be diffusion according to Fick's law. The second is thought to result from partial relaxation of swelling stress. The second slow stage is not observed in large changes in water content and it is suggested that relaxation of swelling stress is greatly accelerated. The second slow stage is, however, observed in desorption. W.R.M.

Penetration and Supercontraction of Keratin Fibres by Lithium Bromide Solutions
A. R. Haly and J. Griffith

Text. Research J., **28**, 32–40 (Jan. 1958)

With the aid of a microscope, measurements were made of the diametral swelling and degree of supercontraction of wool fibres in LiBr soln. at room temperature. Considerable differences were found between supercontractions measured around the orthocortical and around the paracortical sides of the fibres. The mode of penetration of the soln. into the fibres was studied, and it was concluded that the epicuticle is an important barrier to the entry of the reagent. In addition, rates of supercontraction of wool, kid-mohair, and human hair fibres in LiBr soln. at 89° and 100°C. were determined. Supercontraction–time curves exhibit three well defined zones; an attempt is made to explain these characteristics. Some histological observations are reported. S.B.D.

Pyrolysis of Polyamides
S. Straus and L. A. Wall

Bur. Stand. J. Res., **60**, 39–45 (Jan. 1958); *Research Paper* 2819

The pyrolysis *in vacuo* of samples of nylons of mol. wt. ca. 10,000–60,000 has been investigated at temp. of 310–380°C. Rate-of-volatilisation curves showed maxima of ca. 20–40% conversion, corresponding closely with theoretical curves for the random decomposition of a linear polymer. Activation energies calculated from the maxima ranged from 14 kcal., for low-mol.wt. 6,6–6-nylon copolymer to 34 kcal. for high-mol.wt. 6-nylon. The rate of volatilisation is increased by addition of acidic polymerisation catalysts (e.g. H_2SO_4, H_3PO_4) and decreased by free-radical inhibitors (e.g. 1:5-diaminoanthraquinone). It is proposed that both free-radical and ionic mechanisms of decomposition are operating, the ionic mechanism leading firstly to hydrolysis by virtue of water mol. tightly bound to the amide links. This could account for the lower values for activation energy if hydrolysis were rate-determining. The presence of large amounts of CO_2 in the volatile products is also thought to be due to the ionic mechanism. A.J.

Polycaproamides obtained from ε-Caprolactam by the Action of Amine Salts
W. Dawydoff

Faserforsch. und Textiltech., **9**, 61–67 (Feb. 1958)

Polycaproamides prepared from ε-caprolactam using different amino-salts as initiators are characterised by

relative viscosity in conc. H_2SO_4 and the elongation and tensile strength of slivers. Initiation of polymerisation is influenced by dehydration within the initiator and by the number of methylene groups in it. The rate of growth of chains also depends on the initiator. W.R.M.

Cold Drawing of Polyethylene Terephthalate
S. Newman
J. Polymer Sci., **27**, 563–566 (Jan. 1958)

The shoulder separating the drawn and undrawn zones is at an elevated temperature. Estimates give a maximum shoulder temperature of 80°c. At different ambient temperatures the maximum shoulder temperature is approximately constant, and the natural draw ratio tends to unity at 80°c. W.R.M.

Influence of the Scale Factor on the Properties of Synthetic Filaments
A. A. Konkin, V. E. Kotina, and N. V. Demina
Tekstil. prom., **17**, 20–23 (Aug. 1957)

Scale effects are characteristic of fibres and films; e.g. in the case of the glass fibre the relative strength increases with decreasing diameter. In the case of Nitron (polyacrylonitrile) the breaking length increases with increasing filament count, but under the action of ultraviolet radiation the reverse effect is observed, owing to a greater specific surface. Certain physical properties of Nitron fibres coagulated (*a*) in synthetic fatty alcohols, (*b*) in acids, are compared, and the properties are shown to depend on the cross-sectional shape of the fibre. G.J.K.

PATENTS

Coloured Cellulose Acetate Fibres
Lansil *BP* 792,705

Cellulose acetate yarns which have been made by a wet-spinning process and have not been dried, can be dyed rapidly with a wide range of dyes. The dried yarns have good fastness to light and gas-fume fading. W.G.C.

Insoluble Polyvinyl Alcohol Threads
Wacker-Chemie *BP* 778,201

Water insoluble threads of polyvinyl alcohol are produced by treating the dried thread with an aqueous solution, preferably at the boil, containing formaldehyde and hydrochloric or perchloric acid. W.G.C.

Regenerated Protein Fibres
Courtaulds *BP* 778,257

The resistance to water of regenerated protein threads which have been hardened by treatment with an ionogenic cpd. giving rise to cyanate ions is improved by subsequent treatment with an aqueous solution of salts of the metals zinc, cadmium, beryllium, aluminium, zirconium and titanium.

BP 778,258

The hardened threads, before treatment with the solution containing cyanic ions, are treated with an aqueous solution containing a chromium salt. W.G.C.

Reflectance Measurements on Dyed and Undyed Viscose Rayon Fabric in the Ultraviolet and Visible Regions (VIII p. 498)
Fibres from Solutions of Cellulose Xanthates of Low Degree of Substitution (XI p. 502)
Constants in the Intrinsic Viscosity–Molecular Weight Equation (XIII p. 502)
Action of X-Rays on Polyamides (XIII p. 502)
Rendering Pigments, Fibres and Other Fine Particles capable of being readily compounded with Organic Substances (XIII p. 503)
Compressional and Absorptive Behaviour of Bulk Fibre Systems (XIV p. 505)
Sorption of Moisture by Polymers. I—Effect of Crystallinity (XIV p. 505)

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Carbonising Investigations
W. G. Crewther and T. A. Pressley

IV—Industrial Carbonising Trials to assess the Protection of Wool by Surface-active Agents added to the Acid
Text. Research J., **28**, 67–72 (Jan. 1958)

In a series of trials, the addition of a non-ionic surface-active agent to the sulphuric acid bowl resulted, in each experiment, in an increased yield of carbonised wool and, on spinning, increased yarn strength. In a similar trial with combing wool, the addition of a non-ionic surface-active agent to the acid resulted in improved gilling and combing. The amount of acid taken up by the wool was increased by this addition, whereas the water content was decreased; this facilitated drying of the acidified wool.

V—Effect of Water Content on the Action of Sulphuric Acid on Wool: the Significance of Tests for Damage
Ibid., 73–77

The nature of the reaction of sulphuric acid with wool at 105°c. depends on the water content of the acid–wool system. When wool moistened with dil. H_2SO_4 is heated in a closed system to prevent water loss, there is a slow decrease in tensile strength and a marked increase in alkali solubility and, after alkaline pretreatment, in digestibility by trypsin. By contrast, when the acid–wool system is dehydrated before heating, there is a rapid decrease in tensile strength but little change in alkali solubility or trypsin digestibility. The addition of a surface-active agent to the acid prevents loss of strength of the wool, particularly under anhydr. conditions, and it also reduces deterioration of colour. It is concluded that sulphuric acid can attack wool by at least two different reactions and that there is little direct relationship between alkali solubility and fibre strength. S.B.D.

Yellowing during Washing of Viscose Rayon Modified by Oxidation
P. Rochas and L. Gavet
Bull. Inst. Text. France (69), 85–92 (Aug. 1957)

Samples of viscose rayon, modified by various oxidising treatments, were subjected to the following washing procedures—(*a*) soap at 40°c.; (*b*) soap and sodium carbonate at 60°c.; and (*c*) soap and sodium carbonate at 95°c. Aldehydic oxycelluloses showed considerable yellowing and loss of strength after treatments (*b*) and (*c*). Ketonic oxycelluloses suffered a loss in strength without accompanying yellowing. Oxycelluloses containing carboxyl groups were unaffected. Bleaching with sodium chlorite, which does not generate aldehyde groups, gives whites which are most permanent under alkaline washing conditions. Addition of an oxidising agent, such as sodium perborate, to the alkaline wash liquors prevents yellowing of aldehydic oxycelluloses. Whiteness of yellowed samples can be restored by a subsequent bleaching treatment. J.C.F.

Reduction in the Time of Scouring as a result of Changes in the Composition of the Scouring Liquor
T. N. Afanas'eva, S. A. Vvedenskii, and A. S. Stepanov
Tekstil. prom., **17**, 34–36 (Sept. 1957)

By increasing the Na_2SiO_3 content (10 g./litre) in the pressure kier it is possible to reduce the time of scouring, from 8 to 4 hr. for light- and medium-wt. cotton fabrics and from 10 to 4 hr. for coarse calico, without impairing the efficiency of the scour. This results also in a 10–30% saving in caustic soda. G.J.K.

Solo-matic Bleaching System
T. E. Bell
Dyer, **118**, 616 (11 Oct. 1957)

The system is particularly applicable to cotton–synthetic fibre blends. After a light scour in a non-caustic liquor, the cloth is impregnated with an alkaline soln. of H_2O_2 (silicate, caustic soda, and borate), then heated to 204–206°F., and stored for 75 min. in an improved type of J-box which minimises mechanical handling of the cloth. Washing and drying complete the process. A.J.

Improved Method of Wool Washing
V. N. Il'in, S. S. Nazarov, I. B. Frenkel', S. N. Pelevin, and I. N. Preobrazhenskaya
Tekstil. prom., **17**, 46–49 (Dec. 1957)

Woollen and worsted cloths are more readily cleaned using water under pressure in a specially constructed machine, the details of which are given. Improvements cited include a 50–60% reduction in washing time for woollens and 23–43% for worsteds. G.J.K.

Continuous Scouring and Washing of Worsted Dress Material
F. I. Raĭkhlin and M. I. Antonova
Tekstil. prom., **17**, 43–45 (Oct. 1957)

Continuous scouring and washing is recommended for all worsted dress material, the time not exceeding 5 min. in neutral or weakly acid soln. (pH 5·5) at 92–95°c. G.J.K.

PATENT

Reducing the Ash Content of Cellulose
Columbia Cellulose Co. *BP* 792,007

The ash content of cellulose is reduced by washing with an aqueous solution of aldohexonic acid or a lactone or salt of such an acid, e.g. glucono-δ-lactone. C.O.C.

VIII—DYEING

Infrared Properties of Dyes
E. Ganz

Dyer, **119**, 167–172 (31 Jan. 1958)

Translation of paper in *Textil-Rund.*, **12**, 425–432 (Aug. 1957) and *Teintex*, **22**, 661–673 (Sept. 1957). See J.S.D.C., **74**, 65 (Jan. 1958).

Preparation of Coarse Calico and Viscose Rayon Staple Twill for Level Dyeing
S. N. Yablokova

Tekstil. prom., **17**, 42–43 (Dec. 1957)

Prior to dyeing with sulphur dyes to grey and blue shades the loom-state cloths are passed in open width through a two-bowl mangle for impregnation with strong caustic and a wetting agent (4 sec. at 80°C.), a J-box (10–15 min. at 98–99°C.), and a five-box washing machine (81 sec. at 70°C.). The process, which is continuous, is followed by drying. G.J.K.

Reflectance Measurements on Dyed and Undyed Viscose Rayon Fabric in the Ultraviolet and Visible Regions
Ya. A. Legkun

Tekstil. prom. **17**, 67 (July 1957)

Reflectance measurements on undyed viscose rayon fabric in the ultraviolet show considerable selectivity. The reflectance spectra from dyed fabrics are characterised by the presence of two maxima, one in the ultraviolet and one in the visible region, and the corresponding figures for samples dyed with Direct Sky Blue and for those subsequently aftertreated with the fixing agent DTsM are given. When the infrared region is approached, the reflectance of all dyeings increases sharply. More extensive studies of the fading effects by means of the reflectance technique are recommended. G.J.K.

Compilation of Recipes for the Dyeing of Cloth
M. V. Paryšheva

Tekstil. prom., **18**, 45–48 (Feb. 1958)

In a mixture of known dyes the respective quantities of the dyes required for a certain hue can be calculated by correlating reflectance measurements from the dyed cloth with the three-colour system by an empirical formula. Two-dye mixtures have been applied to wool (acid dyes) and cotton (directs), and good results are claimed. G.J.K.

Cibacron Dyes
W. Widmer, M. Peter, and H. Werdenberg

Dyer, **118**, 579–583 (11 Oct. 1957)

A review of methods of dyeing and printing with Cibacron (Ciba) dyes. A.J.

Tendering of Cotton dyed with Sulphur Black
N. D. Mal'tsev and O. I. Agapova

Tekstil. prom., **17**, 37–40 (July 1957)

Molecular sulphur separated on the fibre during dyeing through the decomposition of polysulphides or hydrosulphite does not yield sulphuric acid under the conditions of drying and prolonged storage; rather oxidation of labile atomic sulphur formed through partial decomposition of the dye is held responsible for the formation of sulphuric acid. The simultaneous presence of sulphonic acids is explained by oxidation of thiol groups. Cotton fabric dyed with Sulphur Black responds favourably to aftertreatment with urea or a weak solution of a basic dye. G.J.K.

Tendering of Cotton dyed with Sulphur Black
Z. I. Sergeeva and I. L. Khmel'nitskaya

Tekstil. prom. **18**, 34–37 (Jan. 1958)

The tendering effect of Sulphur Black on cotton is explained on the basis of oxidation of labile polysulphide and disulphide groupings in the dye molecule leading to the formation of peroxy compounds and free sulphuric acid, which, in turn, catalyse further decomposition of the dyed fabric under the action of moisture and oxygen from the air. Decomposition of the dye and tendering of the dyed fabric are prevented by alkylation of the leuco dye on the fabric with excess methyl iodide (10 g. per 30 g. fabric) in an alkaline medium (1% solution) at 30–35°C. Alkylation of the fabric, although yielding slightly redder blacks, does not impair the depth and fastness properties. G.J.K.

Fixing Dyes on the Fabric
N. E. Fedorova and A. N. Vorob'eva

Tekstil. prom., **18**, 42–45 (Feb. 1958)

A new method for dyeing or printing direct dyes and aftertreatment with a fixing agent has been evolved which eliminates padding of the fixing agent and previous drying. The fixing agent, DTsU (amine salt), is used in conjunction with $CuSO_4$. G.J.K.

Improved Colour Effects on Satins
A. A. Morozov

Tekstil. prom., **18**, 55–56 (Feb. 1958)

The technique of the azoic dyeing of satins without drying the cloth after its impregnation with the coupling component is described, and an improved (20–30%) colour intensity compared with the "dry" method is claimed. G.J.K.

Henna Leaves (C.I. Natural Orange 6) for Dyeing Textiles
S. S. Rehsi and E. D. Daruvala

J. Sci. Ind. Research (India), **16A**, 428 (1957): *Chem. Abs.*, **52**, 3347 (25 Feb. 1958)

Henna dyes wool brown from an acid bath. Aftertreatment with chrome turns it reddish brown, with $FeSO_4$ dark brown, $SnCl_2$ brownish yellow, and alum light brown. On non-mordanted cotton a grey is obtained. The dyeings are moderately fast to washing, chlorine bleaching, and light. The leaves yield 1·122% dye. C.O.C.

Action of Nitrous Acid on Proteins
I. Rusznák and A. Pataki

Magyar Kém. Folyóirat, **60**, 282–283 (1954): *Chem. Abs.*, **52**, 3349 (25 Feb. 1958)

When proteins, e.g. wool or silk, are treated with HNO_2, one or more of the following occur: aliphatic amines are deaminated, the tyrosine ring is diazotised, the keratin and fibroin hydrolyse, and HNO_2 (together with HNO_3 formed by oxidation of HNO_2) become attached to the protein molecule. Compounds that react with HNO_2 also react with proteins treated with HNO_2. Protein containing HNO_2 can be dyed by treatment with amines, naphthols, diamines, aminonaphtholsulphonic acids, phenols, etc. With amines it is attributable to the fact that the proteins yield all or part of their attached HNO_2 to diazotise the amines. The diazotised amines then couple with themselves or with the non-diazotised amines to form diazonium compounds capable of combining with the proteins. The depth of the dyeing depends upon the amount of HNO_2 present in the proteins. In the case of the other compounds mentioned both HNO_2 and HNO_3 participate in the reaction, the HNO_2 by diazotising and the HNO_3 by oxidising; both depth and hue of the dyeing depend on the amounts of the two acids present and the protein being treated. C.O.C.

Automatic Temperature Control in Silk Dyeing
V. D. Khokhlov

Tekstil. prom., **17**, 36–39 (Sept. 1957)

Apparatus for automatic temp. control and recording based on the manometric principle is described in detail. Its use in silk dyeing is recommended. G.J.K.

Monolayers. VII—Reactions of Casein with Dyes and other Aromatic Solutes and their Relation to Adsorption by Protein Fibres
A. Cameron, C. H. Giles, and T. H. MacEwan

J.C.S., 1224–1230 (March 1958)

The effects of aromatic sulphonates, including some dyes, upon casein monolayers, spread on acid or on buffer soln. near the isoelectric point, are found to be as follows—Large dye mol. with a non-ionic polar group at each end have an effect on the film similar to that of tannic acid, and surface-active monobasic dye mol. with weak hydrogen-bonding groups penetrate the film and at high surface pressures increase its solubility. The latter effect may be due to the adsorption of a layer of dimerised dye mol. below the film. Small sulphonate mol. and disulphonates with weak hydrogen-bonding centres (e.g. anthraquinonedisulphonates) are probably adsorbed beneath the film. The results are used as a basis for the hypothesis that the affinity of monobasic anions for protein fibres arises from

their own mutual attraction, which assists them in forming a monolayer or a layer of micelles adsorbed on the fibre, rather than from specific anion–fibre attraction.

H.H.H.

Diffusion of Acid Dyes in Polyamide Fibres

T. Shimizu and U. Miyaoka

Sen-i Gakkaisha, **13**, 553–559 (1957): *Chem. Abs.*, **52**, 2412 (10 Feb. 1958)

The diffusion coefficient of 3 acid dyes (C.I. Acid Red 13, 27, and 88) on Amilan fibres when calculated by Hill's equation or observed microscopically were about ten times as large with unstretched as with stretched fibres apparently being affected by the fine structure of the fibres. They decreased with higher sulphonation which increased, however, the activation energy of diffusion. The coefficient D in μ^2/min. was found empirically related to the time of half-dyeing as follows— D = denier/$0{\cdot}511t_{0\cdot5}$.

C.O.C.

PATENTS

Dyeings of Extremely Good Wash Fastness on Cellulose Acetate

Celanese Corpn. of America *BP* 791,391

Cellulose acetate, of acetyl value $\nless$ 59% (calculated as acetic acid), is dyed in an aqueous bath with a disperse dye, preferably of the high temperature, slow dyeing type, after which the dyed material is heated. Thus cellulose acetate fabric of acetyl value 62·0–62·5% was dyed with 2% of Eastman Fast Blue GLF (C.I. Disperse Blue 27) at 85°C. using as assistants tri-*n*-propyl phosphate and Igepon T. The dyed material was then heated under tension at 230°C. for 1 min. The resulting dyeing was unaffected by three repetitions of the AATCC No. 3 wash fastness test.

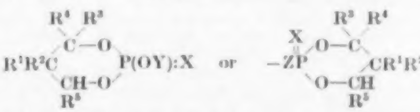
BP 791,392

Dyeings of very good fastness to washing are quickly obtained with disperse dyes in presence of an assistant which is a solvent for the dye and is substantive to cellulose acetate, e.g. tri-*n*-propyl phosphate. C.O.C.

Dyeing Polyacrylic Fibres

Union Carbide Corpn. *BP* 791,531

Fibres containing acrylonitrile polymers or copolymers can be dyed to yield high colour values with both water soluble and disperse dyes, e.g. acid, disperse, and vat dyes, when dyeing is carried out in presence of 0·2% (on wt. of fibres) of compounds of formula—



(R^1, R^2 R^3, R^4 and R^5 = H or Alk of 1–8 C; X = O or S; Y = Alk, Ar, alkaryl, halogeno alkyl, or cyanoalkyl; Z = divalent dioxyalkylene residue of an alkylene or polyalkylene glycol or of a dihydricphenol or diphenylolalkane) e.g. 5-ethyl-2-(2-ethylhexyloxy)-2-oxo-4-propyl-1:3:2-dioxaphosphorinane. C.O.C.

Dyeing of Polyester Fibres and Films

ICI *BP* 792,210

The fibres or films are impregnated with a water insoluble dye or with intermediates which can be converted to such a dye *in situ*, and are then exposed to the saturated vapour of one or more halogenated aliphatic hydrocarbons. This gives rapid and effective dyeing. Thus Terylene staple fibre fabric is impregnated with a 3% aqueous dispersion of Dispersol Fast Yellow A 200 Paste (C.I. Disperse Yellow 1) containing 1% of gum tragacanth and 1% of the Na salt of a highly sulphonated oil. It is then immersed in the vapour of boiling trichloroethylene for 3 min., plunged into boiling water for 30 sec. and finally washed for 20 min. in oleyl sodium sulphate and soda ash. This yields a full yellow which is fast to rubbing.

C.O.C.

Clear and Pigmented Oil-in-Water Resin Emulsions (III p. 492)

Relation Between the Absorption Spectra and the Chemical Constitution of Dyes. XXIX— Interaction of Direct Azo Dyes in Aqueous Solution (IV p. 492)

Coloured Cellulose Acetate Fibres (VI p. 497)

Printing and Pad-dyeing of Polyacrylonitrile Fabrics (IX p. 500)

Sodium Carboxymethylcellulose of Low Degree of Substitution and its Properties as a Finish for Textiles (XI p. 502)

Mode of Action of Fluorescent Brightening Agents and Measurement of their Relative Efficiency (XIV p. 504)

Study of Dyes and Fibres during Fading— Chemical Transformation of *p*-Aminoazobenzene by Ultraviolet Radiation (XIV p. 507)

Estimation of Acidity in Cellulose — Reaction of Crystal Violet Base (C.I. 42555B) with Cotton (XIV p. 507)

IX—PRINTING

Humidification of Cotton prior to Steaming

N. A. Oskorbina and F. I. Sadov

Tekstil. prom., **17**, 41–42 (July 1957)

The moisture content of the fabric prior to steaming is correlated with the time of steaming in printing using three vat dyes (Thioindigo Orange KKh, Thioindigo Scarlet, and Bright Green Zh). The application of a very fine water spray to the back of the fabric (yielding a 20–30% moisture content) is recommended immediately prior to placing the fabric in the ager. G.J.K.

Improved Fastness with Azoic Dyes

V. E. Rostovtsev

Tekstil prom., **18**, 48–50 (Feb. 1958)

The fastness towards light, perspiration, and wet and dry rubbing of insoluble azo dyes (red, bordeaux, and blue) in printing is found to be superior when the diazotised amine is coupled with the azoic coupling component Azotol PA as compared with Azotol A. On the other hand, although Azotol PA imparts greater substantivity than does Azotol A, these two components can be washed out from the fibre with equal ease. G.J.K.

Optimum Conditions for Printing Viscose Rayon Staple Fabric with Diazol Blue O

F. I. Sadov and N. N. Voznesenskii

Tekstil. prom., **17**, 31–34 (Dec. 1957)

The optimum conditions for printing of viscose staple fabric with Diazol Blue O are established by varying the pH and the ratio of the diazol to the azoic coupling component; pH is measured in the printing paste and in the fabric after coupling. The importance of the conditions of impregnation of the fabric with the azoic coupling component is stressed. Introduction into the dye paste (prepared with lactic acid) of $ZnSO_4$ or $MgSO_4$ (8–9 g./litre) increases pigment formation on the fabric by 7–8%. Good fastness to drastic washing at the boil and to rubbing (dry and wet) are claimed. G.J.K.

Formation of Phthalocyanine Dyes on Cotton

Bao Chi-min

Tekstil. prom., **17**, 52–54 (Dec. 1957)

Various methods for the preparation of phthalocyanine dyes in the printing paste and on the fibre are reviewed.

G.J.K.

Damage caused in Cellulose by Treatment with the Neutral System Sodium Formaldehyde-sulphoxylate–Hydrogen Peroxide

I. Rusznák and O. Boros

Deut. Textiltech., **9**, 522–523 (1957): *Chem. Abs.*, **52**, 3228 (25 Feb. 1958)

A study of the oxidation of Rongalite by Ludigol (Na *m*-nitrobenzenesulphonate) and by H_2O_2. Rongalite is completely oxidised by H_2O_2 with formation of bisulphites. The reaction is quick, strongly exothermic and causes fibre damage in discharge printing. Ludigol oxidises Rongalite only to S compounds of lower oxidising potential. The decrease in pH caused by H_2O_2 either in Rongalite solutions or printing pastes can be largely prevented by addition of $CaCO_3$ or Pb acetate with favourable results, as these replace the SO_4 by weak acids and so result in less degradation of the fibre. C.O.C.

Energy Transfers between Crystals and Adsorbed Dyes. I— Desensitisation

R. C. Nelson

J. Opt. Soc. Amer., **48**, 1–3 (Jan. 1958)

Some photographically desensitising dyes are shown to desensitise also photoconductivity in cadmium sulphide films, and are considered to be capable of acting as surface electron-traps. Dye electron affinities, in the region of

3·5 e.v., are much lower than those of the sensitive specks on the silver bromide grains. R.B.B.

PATENTS

Printing and Pad-dyeing of Polyacrylonitrile Fabrics
S *BP* 791,294

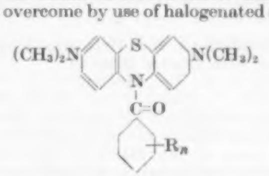
Use of an aqueous printing paste or padding liquor containing a water-soluble organic dye, a compound yielding acid on heating or a non-volatile acid, and cuprous oxide or a water-soluble cupric compound together with an inorganic reducing agent, followed by drying and steaming enables the process described in *BP* 767,053 (J.S.D.C., **73**, 279 (1957)) to be used for the printing and pad-dyeing of polyacrylonitrile fabrics. C.O.C.

Diazotype Material
Charles Bruning Co. *USP* 2,780,547

Diazotype material which yields prints of even, non-grainy character and good dye density is obtained by treating the base, e.g. sized paper or cloth, before or simultaneously with the application of the sensitising solution, with an aqueous dispersion of non-cooked and non-swollen rice starch at < 125°F. C.O.C.

10-(Halo-benzoyl)-3:7-bis(dimethylamino)phenothiazines for Use in Copy Printing
National Cash Register Co. *BP* 791,426

The colourless benzoyl *leuco*methylene blue (10-benzoyl-3:7-bis(dimethylamino)phenothiazine) is used in coating compositions for manifold record sheets. Colour is developed by contact with acid clays which may be incorporated in the copy-paper. The disadvantage of the above *leuco*-compound is that it gradually decomposes on exposure to light to give an undesirable blue-green coloration to the manifold record sheet. This difficulty is substantially overcome by use of halogenated derivatives—



(R = Cl or Br; n = 1 to 5). These are made, e.g. by adding the $ZnCl_2$ double salt of *leuco*methylene blue (73) to pyridine (122) and then adding o-chlorobenzoyl chloride (50) slowly at 90°C. followed by refluxing for 60–90 min. Alternatively, mixed halogeno-benzoyl chlorides are made by passing a halogen gas through benzoyl chloride in presence of a catalyst, e.g. Fe and iodine and these products are then reacted with the $ZnCl_2$ double salt of *leuco*methylene blue as before. E.T.

Dispersions of Colour Couplers for Use in Photographic Silver Salt Emulsions
Kodak *BP* 791,219

Method for preparing dry low ratio colour coupler dispersions containing no silver halide but which are readily miscible with gelatino-silver halide emulsion.

BP 791,353

Dispersions of colour couplers in gelatin emulsions, the dispersions having a low ratio of oil former to coupler and showing a minimum of coupler crystallisation are prepared by making two separate dispersions—(1) a dispersion of the colour coupler in gelatin in absence of a solvent, and (2) a dispersion of the oil former alone in gelatin. The two dispersions are then added to a gelatino-silver halide emulsion before it is used for coating. C.O.C.

Subtractive Colour Film
E. Gretener *BP* 791,675

Method for producing subtractive colour film containing a black-and-white recording, the transparency of which is equal at every image point to the greatest of the three colour components, combined with a subtractive dye recording, which at every image point contains only two subtractive dyes, thus resulting in a linear recording of colour components.

BP 791,676

An adaptation of the above process to meet the requirements of coloured cinema film. C.O.C.

Electrostatic Printing
Horizons *BP* 792,708

A method of using the electronically produced image on the face of a cathode ray tube for printing purposes. C.O.C.

Thickening Agent for Printing with Vat Dyes (III p. 491)
Clear and Pigmented Oil-in-Water Resin Emulsions (III p. 492)
Nitro-derivatives of Benzoyl *leuco*-Methylene Blue Transfer Coatings for Manifold Copy Sheets (IV p. 494)
Resin-bonded Pigment Dispersions (V p. 496)
Cibacron Dyes (VIII p. 498)

X—SIZING AND FINISHING

Physical Properties of Chemically Modified Cottons IV—Effects of Partial Carboxymethylation
J. N. Grant, A. W. McDonald, and G. C. Humphreys
Text. Research J., **28**, 60–66 (Jan. 1958)

Yarns from six cottons selected for their widely different inherent fibre characteristics were partially carboxymethylated to a degree of substitution averaging about 0·125 while held at their original length. Moisture regain, linear density, breaking load, and elongation at break were increased by the modification. Fibre cellulose density and length decreased. Samples of the different cotton varieties were found to differ in their response to the treatment with interrelationships existing between the linear density, degree of substitution, and moisture regain. Changes in fibre properties in the caustic treatments of cottons, such as carboxymethylation and mercerisation, were found to be similar when the tensional conditions during treatment were essentially equal. S.B.D.

PATENTS

Yarn-finishing Process
ICI *BP* 777,992

Loose fibres in yarn spun from synthetic thermoplastic filaments, e.g. nylon and Terylene, can be eliminated in a manner similar to singeing, by passing the yarn through an aperture slightly larger than the diameter of the yarn, the material forming the aperture being heated to 350°C. W.G.C.

Antistatic Finish
N.V. Onderzoekingsinstituut Research *BP* 791,476

An antistatic finish is imparted to textiles and moulded articles made from synthetic hydrophobic polymers, e.g. nylon or polyesters, by treating them with an aqueous solution of a polymer containing several carboxylic or sulphonic acid groups and which loses its solubility in water when heated to > 100°C., e.g. polyacrylic acid, drying and then heating them to > 100°C. C.O.C.

Rotproofing of Cellulosic Materials
National Cylinder Gas Co. *BP* 791,105

Cellulosic material is impregnated with copper formate to give a Cu content of 0·01–2·0% and then heated to > 100°C. until the green imparted by the copper formate has changed to olive or chocolate brown. C.O.C.

Dimensional Stabilisation of Cellulosic Fabrics by applying Alkyl Silicon Halide Vapours
E. Robbart *USP* 2,782,090

The fabric is treated with a gaseous mixture, preferably an aerosol, of an organohalogeno silane and an inert gas at < 100°F., preferably < 80°F. for 1 sec.–3 min., then immediately neutralised by treatment with a mild alkaline solution and dried under tension, preferably weftways tension. C.O.C.

Reducing the Felting Power of Wool while increasing its Abrasion Resistance
Rohm & Haas Co. *USP* 2,780,567

Much reduction in the shrinking and felting properties of textiles containing wool is obtained by treatment with an aqueous dispersion of a water-insoluble, linear polymer containing 3% of a monomer of formula—

$$CH_2{:}CH{\cdot}A{\cdot}R^1{\cdot}OH$$

(A = O or S; R^1 = straight or branched chain alkylene of 2–10C, $C_2H_4AC_2H_4$, $C_2H_4(R^2)C_2H_4$ or $C_2H_4N(R^3)(R^4)(X)$-C_2H_4 (R^2 = H, CH_3, C_2H_5 or C_2H_4OH; R^3 and R^4 = CH_3, C_2H_5 or C_2H_4OH; X = a negative radical). A suitable polymer is the copolymer of diethyleneglycol monovinyl ether with butyl acrylate.

USP 2,780,608

An aqueous dispersion of a water-insoluble linear polymer of monoethylenically unsaturated molecules comprising $\not<$ 3% by wt. of 3-chloro-2-hydroxypropyl acrylate or methacrylate is used. C.O.C.

Water-repellent and/or Flame-resistant Finish

A. M. L. de Luxembourg *USP* 2,780,566

Fibrous material is treated with an aqueous mixture of aluminium sulphate, lead acetate and hexamethylene tetramine and then exposed to infrared radiation. This yields a water-repellent finish which can be made flame-resistant by giving the material a further treatment with a soluble tin salt and again exposing it to infrared radiation. The treated material is fast to repeated laundering or dry-cleaning and is unaffected by exposure to light. C.O.C.

Flame-resistant Finish

Ciba *USP* 2,781,281

Textiles are rendered flame-resistant without loss in tensile strength or handle by impregnating them with an aqueous liquid containing (1) 12–48% by wt. of the product obtained by heating excess urea with ortho-, pyro- or meta-phosphoric acid or an ammonium salt of one of these acids, and (2) 2–18% by wt. of a methylol compound of an amino triazone, the ratio of component (1) to (2) being 3:1 to 6:1, and finally baking the impregnated fabric. C.O.C.

Improving the Handle of Polyester Fibre Fabric

DuP *USP* 2,781,242

The fabric is subject to dry heat while relaxed and is then treated with an aqueous hydroxide of an alkali metal or salt thereof derived from a weak acid. Thus fabric of continuous filament polyethylene terephthalate was heated while relaxed on a tenter at 210°C. for 2 min. and then treated for 15 min. in boiling 5% aq. NaOH. It lost 14% in weight but had a much more pleasing handle. C.O.C.

Wrinkled or Rippled Finish

Bradford Dyers' Assocn. *BP* 791,553

Compressively-shrinkable sheet material, e.g. cloth, leather, plastic sheeting or paper, is passed, together with a second sheet material which buckles, through a compressive shrinking machine. C.O.C.

Permanent Embossing of Thermoplastic Fabrics

Decora Corpn. *USP* 2,783,175

An impervious endless belt of heat-resistant plastic, the fabric to be embossed, an endless patterned web, e.g. fish net, and an endless metallic belt are fed together in this order, the plastic belt being uppermost, through a pair of squeeze rollers. The metallic belt is perforated with 0·125 in. holes with their centres 0·75 in. apart. The assembly then passes over a suction box and at the same time passes under a radiant heating element. Finally the assembly passes between a pair of squeeze rollers and the impervious belt and the metallic belt are removed. The fabric still in conformity with the patterned web then passes over a series of chilled rolls, and then over several festoon rolls, and through the nip of a pair of chilled rolls. Finally the patterned web is taken away and the embossed fabric batched. C.O.C.

Flock-coated Textile Fabrics

Heberlein & Co. *BP* 791,797

A base fabric having high elasticity in length and/or width is coated with an adhesive and then with flock. After the adhesive is dried the fabric is stretched in length and/or width to produce tears in the layer of adhesive. This restores the air-permeability of the base fabric without materially affecting the adhesion of the flock to it. C.O.C.

Colour Effects on Textiles

Heberlein & Co. *BP* 792,072

Cloth is exposed in a high vacuum to metal vapour and then to the vapour of stannic oxide, antimony trioxide or lead oxide. The colour of the treated cloth varies according to the thickness and the refractive index of the deposited metal oxide and with the angle at which it is viewed. C.O.C.

Pyrolysis of Polyamides (VI p. 496)
Rendering Organic Derivatives of Cellulose More Receptive to Printing Inks and Other Coatings (XI p. 502)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Polarised Infrared Spectra of Cellulose I

J. Mann and H. J. Marrinan

J. Polymer Sci., **27**, 595–596 (Jan. 1958)

Tsuboi's work (*ibid.*, **25**, 159 (1957)) on the O–H or O–D stretching vibrations is discussed. The band at 3309 cm.$^{-1}$ is found to be perpendicular to the fibre axis, while Tsuboi reports the corresponding O–D band to be parallel. W.R.M.

Transformations of Cellulose at Elevated Temperatures. VI— Effect of the Addition of Glucose on the Formation of Laevoglucosan during Thermal Decomposition *in vacuo*

O. P. Golova, A. M. Pakhomov, and E. A. Andrievskaya

Izvestiya Akad. Nauk S.S.S.R. otdel khim. nauk, 1499–1500 (Dec. 1957)

The addition of glucose (2–20%) during the thermal decomposition of cellulose *in vacuo* causes a 50% decrease in the yield of laevoglucosan. G.J.K.

Mechanism of the Periodate Oxidation of D-Glucose

F. S. H. Head

Chem. and Ind., 360–361 (22 March 1958)

Data are given for the oxidation of 0·025 M. D-glucose with 0·1375 M. sodium metaperiodate at 20°C., which afford some support to recent work by Warsi and Whelan (*ibid.*, 71 (1958)), and confirm the view that D-glucose is rapidly oxidised to 2-*O*-formyl-D-glyceraldehyde, with reduction of 3 moles of periodate and liberation of 2 moles of free acid, formaldehyde being released only after hydrolysis of the ester has occurred (cf. also Karrer and Pfaehler, *Helv. chim. Acta*, **17**, 766 (1934)). H.H.H.

Alkaline Degradation of Polysaccharides. IV— Monosaccharide Analogues of Periodate Oxycellulose

D. O'Meara and G. N. Richards

J.C.S., 1204–1211 (March 1958)

The products of alkaline degradation of certain periodate-oxidised monosaccharides indicate the degradation to occur predominantly by scission of the carbon–oxygen linkage in the β-position with respect to the aldehyde groups. Subsequent rearrangements of the products appear to yield a mol. of glycollic acid from each scission of this type. In certain circumstances an intramolecular Cannizzaro rearrangement can occur in the periodate-oxidation product. The analogy with periodate oxycellulose is discussed. H.H.H.

Alkaline Degradation of Periodate-oxidised Polysaccharides

D. O'Meara and G. N. Richards

Chem. and Ind., 40 (11 Jan. 1958)

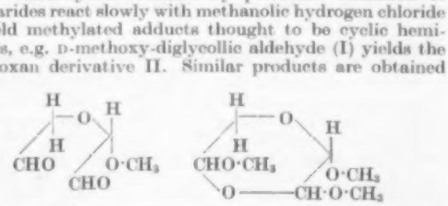
On treatment with oxygen-free lime-water, model compounds of periodate-oxidised polysaccharides containing a β-alkoxy-carbonyl system, undergo mainly an elimination reaction to yield a glyoxal derivative, which rapidly rearranges to glycollic acid and an alcohol; and a second intermediate product which yields a butyric acid derivative. Decomposition via a Cannizzaro reaction is relatively unimportant. A.J.

Reaction of Periodate-oxidised Polysaccharides with Alcoholic Hydrogen Chloride

I. J. Goldstein and F. Smith

Chem. and Ind., 40–42 (11 Jan. 1958)

The dialdehydes obtained by periodate oxidation of saccharides react slowly with methanolic hydrogen chloride to yield methylated adducts thought to be cyclic hemiacetals, e.g. D-methoxy-diglycollic aldehyde (I) yields the 1:4-dioxan derivative II. Similar products are obtained



(I) (II)

from periodate-oxidised methyl α-D-glucopyranoside, methyl 4:6-*O*-benzylidene-α-D-glucopyranoside, and methyl 6-*O*-methyl-α-D-galactopyranoside. Periodate-oxidised polysaccharides, e.g. from amylopectin, amylose, corn starch, and cellulose, also react with methanolic hydrogen chloride yielding insol. methylated hemiacetals, which are

thought to be cross-linked. There is a linear relation between the degree of periodate oxidation and the methoxyl content of the final products. A.J.

Fibres from Solutions of Cellulose Xanthates of Low Degree of Substitution
E. A. Abramova, S. L. Munte, and N. K. Shemkov

J. Appl. Chem. U.S.S.R., **30**, 1815–1820 (Dec. 1957)

A dry, sandy, deep yellow cellulose xanthate was obtained by treatment of alkali cellulose with 15% CS_2 at 8–10°c. for 90 min. and 28°c. for 90 min. When this compound is dissolved in the solvent at −5°c. to give a soln. composed of 7·5–8% α-cellulose and 6·8–6·9% NaOH, a product showing good filtration properties is produced. It is possible to obtain a yarn of good wet strength by extruding the viscose soln. at 58·7 m./min. into the coagulating bath consisting of per litre, 130–140 g. H_2SO_4, 350–375 g. Na_2SO_4, and 17–18·5 g. $ZnSO_4$. The coagulating bath of large sulphate content gives better yarn. The minimum temp. of spinning is 45°c. T.Z.W.

Intrinsic Viscosities of Cellulose Derivatives
P. J. Flory, O. K. Spurr, and D. K. Carpenter

J. Polymer Sci., **27**, 231–240 (Jan. 1958)

Intrinsic viscosities $[\eta]$ of cellulose acetates and cellulose trinitrate have been investigated as functions of temperature, solvent, and molecular weight. Large negative temperature coefficients of $[\eta]$ and variations of $[\eta]$ with solvent are ascribed to changes in chain flexibility. W.R.M.

Thermodynamic Properties of Dilute Solutions of Cellulose Acetate
W. R. Moore and B. M. Tidswell

J. Polymer Sci., **27**, 459–472 (Jan. 1958)

Heats, free energies, and entropies of dilution for solutions of a secondary cellulose acetate in representative solvents are obtained from temperature coefficients of osmotic pressures. All the systems are endothermal. It is possible, if solvation and the stiffness and segment size of the chains are allowed for, that the results may be interpreted in terms of simple lattice theories. W.R.M.

Sodium Carboxymethylcellulose of Low Degree of Substitution and its Properties as a Finish for Textiles
G. A. Petropavlovskii and G. G. Vasil'eva

J. Appl. Chem., U.S.S.R. **30**, 1832–1837 (Dec. 1957)

The carboxymethylcellulose is prepared by treatment of wood cellulose with $Cl{\cdot}CH_2{\cdot}COOH$ at 40°c. for 4 hr. The ripening of the derivative produced for about 40 hr. after etherification decreases the viscosity of its soln. in NaOH, which makes possible to increase its concn. to 6·8%. Solutions of low-substituted carboxymethylcellulose in 6·5% NaOH are stable and can be diluted to 0·5% NaOH content. They are suitable for aftertreating cotton yarns and fabrics, producing fastness to rubbing and washing. The fastness to rubbing is directly proportional to the D.P. of the derivative and the concn. of the soln. and is inversely proportional to the degree of substitution. The presence of a large quantity of finish on the fibre increases its strength. T.Z.W.

PATENTS

Paper having Improved Wet Strength
N. V. de Bataafsche Petroleum Maatschappij

BP 792,946

Pulp or paper is treated at any stage of manufacture with an aqueous medium containing a polyether polyepoxide and an epoxy curing agent, drying and then curing the polyepoxide. C.O.C.

Rapid Setting of Baryta Coatings
Eastman Kodak Co. *USP* 2,780,560

Baryta coatings free from surface roughness are obtained if the paper is first treated with a buffer material of pH7–8 which causes rapid setting of baryta coating composition containing formaldehyde. Thus paper coated with 0·8 g. trisodium phosphate and 0·4 g. citric acid per sq.m. was coated with a composition made up of an aqueous dispersion containing 3% gelatin and 10% $BaSO_4$ and 0·35% of 40% formalin. The coating sets almost immediately. C.O.C.

Rendering Organic Derivatives of Cellulose More Receptive to Printing Inks and Other Coatings
Celanese Corpn. of America *BP* 791,776

Subjecting cellulose acetate film or the like to a corona electrical discharge much improves its receptivity towards printing inks and other coating materials. C.O.C.

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Interaction of Intermediates and Oxidising Agents in the Dyeing of Furs
P. I. Chatskii

Legkaya prom., **16**, (12), 30–34 (1956): *Chem. Abs.*, **51**, 13443 (1957): *J. Amer. Leather Chem. Assocn.*, **53**, 188 (March 1958)

Changes undergone by the Protein Molecule. I—Interaction of the Primary Breakdown Products of Protein with the Anhydrides of *N*-Carboxyglycine, DL-Alanine, and DL-Proline
K. T. Poroshin

Izvestiya Akad. Nauk S.S.S.R., otdel khim. nauk, 996–998 (Aug. 1957)

A series of initiators (e.g. amidines and guanidines) have been used in the polycondensation of protein breakdown products with *N*-carboxyglycine and DL-alanine, and derivatives of casein in the form of compounds such as a polyglycyl hydrolysate of peptone were obtained. G.J.K.

Interaction of Small Ions with Proteins
K. H. Doremus and P. Johnson

J. Physical Chem., **62**, 203–210 (Feb. 1958)

The interaction of sodium, calcium, and phosphate ions with serum albumin has been studied by conductance and transfer experiments. Appreciable sodium binding in basic solutions was found. Calcium ion binding was greater than sodium and reached a limit with increasing calcium concentration. It is suggested that sodium binding is primarily electrostatic. W.R.M.

PATENT

Leather
FBy *BP* 793,603

Modification of *BP* 756,175. Cationic or non-ionic emulsifiers having marked tannin-precipitating properties may be used instead of anionic emulsifiers. C.O.C.

XIII—RUBBER; RESINS; PLASTICS

Constants in the Intrinsic Viscosity–Molecular Weight Equation
A. Sharples and H. M. Major

J. Polymer Sci., **27**, 433–440 (Jan. 1958)

The constants K_m and a in the equation $[\eta] = K_m\bar{M}_v^a$ where $\bar{M}_v$ is the viscosity-average molecular weight and $[\eta]$ the intrinsic viscosity, are usually found from measurements of $[\eta]$ for fractions whose weight-average $\bar{M}_w$, or number-average M_n molecular weights have been determined, assuming that $\bar{M}_v = \bar{M}_n = \bar{M}_w$. It is suggested that, for a distribution arising from random degradation, $\bar{M}_v$ = constant × $\bar{M}_n$. This distribution results from heterogeneous hydrolysis of cotton. Applied to cellulose triacetate in chloroform, the relationship gives $K_m = 2{\cdot}51 \times 10^{-4}$ and $a = 1{\cdot}02$. W.R.M.

Alkylation of ε-Caprolactam and Aliphatic Diamines by Catalytic Dehydration
A. S. Shpital'nyĭ and T. V. Kuznetsova

J. Appl. Chem. U.S.S.R., **30**, 1848–1850 (Dec. 1957)

When a mixture of 45 g. ε-caprolactam and 65 g. CH_3OH at 120°c. is passed through a reactor, filled with 150 g. of a catalyst at 350°c., at a speed of 30 ml./hr., 32·5 g. *N*-methyl-ε-caprolactam was separated from the distillate by fractionation. *N*-Ethyl-ε-caprolactam, methyl and ethyl derivatives of ethylenediamine, and hexamethylenediamine were prepared similarly. Activated Al alum or alumina can be used as catalyst, the latter giving better results. 350°c. is the most suitable temp. T.Z.W.

Action of X-Rays on Polyamides
B. L. Tsetlin and S. R. Rafikov

Izvestiya Akad. Nauk S.S.S.R., otdel khim. nauk, 1411–1413 (Nov. 1957)

The change in the thermomechanical properties of both stretched and unstretched polyamide fibres under the influence of high-intensity X-rays is investigated, and the experimental findings are correlated with the concept of radiation-induced vulcanisation of polyamides and destruction of crystallinity. G.J.K.

Heterochain Polyamides
V. M. Kharitonov, T. M. Frunze, and V. V. Korshak

III—Kinetics of Polymerisation of ε-Caprolactam in the presence of Hexamethylenediamine Adipate
Izvestiya Akad. Nauk S.S.S.R., otdel khim. nauk, 1134–1136 (Sept. 1957)

The polymerisation of ε-caprolactam is a bimolecular process. On the basis of mol.-wt. determinations the rate of growth of the polymer is rapid in the initial stages, but becomes constant owing to hydrolytic breakdown.

IV—Kinetics of the Formation of Mixed Polyamides from Salts of Hexamethylenediamine
Ibid., 1136–1138

An investigation of the polymerisation of mixtures of hexamethylenediamine salts with adipic and azelaic acids shows that initially the adipic acid is the more reactive, but at later stages equilibrium is reached, the polymer having the same composition as the initial monomer mixture. The final stages of the polycondensation are controlled by the proportions of the initial reactants and not by the kinetics of the individual steps. G.J.K.

Heterochain Polyesters
V. V. Korshak, S. V. Vinogradova, and V. M. Belyakov

VI—Polyesters of the 3:3′- and 4:4′-Dicarboxylic Acids of Diphenylmethane and of Benzophenone-4:4′-dicarboxylic Acid
Izvestiya Akad. Nauk S.S.S.R., otdel. khim. nauk, 998–1000 (Aug. 1957)

Polyesters of the title acids with eleven aliphatic glycols of different structure have been synthesised and investigated, and the properties of these polyesters correlated with the structure of the initial components.

VII—Polyesters of *p*-Phenylenediacetic and *cis*- and *trans*-Hexahydroterephthalic Acids
Ibid., 1000–1001

The preparation and properties of the polyesters of these acids with eleven different glycols are described, and the influence of the structure of the glycol, of the *cis* or *trans* configuration of the acid, and of the indirect attachment of the acid group to the aromatic nucleus and the alicyclic ring system upon the properties of the polyesters are discussed. G.J.K.

Melting Points and Heats of Fusion of Polyamides and Polyesters
P. J. Flory, H. D. Bedon, and E. H. Keefer
J. Polymer Sci., **28**, 151–161 (Feb. 1958)

Melting points of three polyamides, four polyesters, and their mixtures with diluents are determined, and heats of fusion calculated from depressions of melting point. Heats of fusion of polyesters exceed those for corresponding polyamides. High melting points are associated with low entropies of fusion rather than with high heats of fusion. W.R.M.

Polymerisation of Acrylonitrile by Boron Trifluoride
T. W. Campbell
J. Polymer Sci., **28**, 87–96 (Feb. 1958)

The activity of boron trifluoride–dimethylformamide as a catalyst for the polymerisation of acrylonitrile is studied. Oxygen is a necessary third component, and polymerisation is a free-radical process. It is suggested that oxygen reacts with acidic complexes of dimethylformamide to give peroxides which act as typical peroxide initiators. W.R.M.

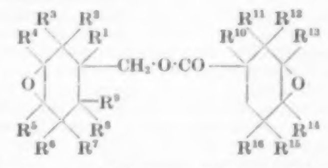
PATENTS

Rendering Pigments, Fibres and Other Fine Particles capable of being readily compounded with Organic Substances
General Tire & Rubber Co. *USP* 2,780,612

The pigments, fibres or the like are treated with a compound of formula R·NCO (R = alkenyl, arylalkenyl, etc.) to render them more readily wettable by and interlocked with polymerisable organic compounds and polymers. Thus Carbon Black (C.I. Pigment Black 7) after being treated with the mixed vapour of methyl and ethyl*iso*cyanates has much less tendency to be wet by water and much greater tendency to be wet by hydrocarbon solvents. Similarly cotton, viscose rayon or glass fibres treated with allyl*iso*cyanate vapour are more readily wetted with polymerisable polyester compositions; fibres so wetted and then moulded and cured have better water resistance and strength than if the fibres had not been treated with the *iso*cyanate. C.O.C.

Plasticisers having Heat and Light Stabilising Properties for Vinyl Halide Polymer Compositions
Union Carbide Corpn. *BP* 793,595

Diepoxides of formula—



(R^1–R^{16} = H or Alk), e.g. 3:4-epoxy-6-methyl*cyclo*hexylmethyl-3:4-epoxy-6-methyl*cyclo*hexanecarboxylate, are used. C.O.C.

Flame-resisting Compositions for Resinous Polymers
Diamond Alkali Co. *BP* 792,368

A chlorinated organic material containing ≮ 50% by wt. of chlorine and ≮ 20% of an aryl substituted phosphate, e.g. a mixture of chlorinated paraffin wax and tricresylphosphate is a good flame-resisting agent for incorporating into resinous polymers. C.O.C.

Pigmented Silicone Elastomers
General Tire & Rubber Co. *USP* 2,780,611

Pigments, e.g. Carbon Black (C.I. Pigment Black 7), Silica (C.I. Pigment White 27) and TiO_2 (C.I. Pigment White 6) are treated with compounds containing no polar groups but capable of reacting with an *iso*cyanate and having, or capable of forming compounds having, > 1 C containing group capable of readily reacting with an alcoholic OH group to form a carbon–oxygen bond, the C atom of that group being connected by 2 valence bases to N or other C atom, e.g. toluene di*iso*cyanate. Silicone elastomers to which the treated pigments have been added in amounts of about 10–200% are readily cured to yield elastic products of tensile strength > 1000 lb./sq. in. C.O.C.

Polycaproamides obtained from ε-Caprolactam by the Action of Amine Salts (VI p. 496)
Antistatic Finish (X p. 500)

XIV—ANALYSIS; TESTING; APPARATUS

Increased Chemical Reactivity of the Surface compared with that in the Bulk Volume of Britton-Robinson Universal Buffers
R. G. Pike and D. Hubbard.
Bur. Stand J. Res., **59**, 411–414 (Dec. 1957); *Research Paper*, 2811

Interferometric examination of pieces of optically flat glass (Corning 015) after partial immersion in phosphate–acetate–borate buffers at 80°c. for 6 hr. revealed some chemical attack of the fully immersed portion and, in the pH range 7–11·8, enhanced attack at the liquid–air interface. This is due to the higher concn. in the surface and the consequent higher surface pH, the difference being as much as 2·4 pH units at pH 7 (in the bulk soln.). The properties of the glass did not allow acidic soln. to be investigated. A.J.

Determination of Small Amounts of Pyrophosphate in Soluble Orthophosphates
W. B. Chess and D. N. Bernhart
Anal. Chem., **30**, 111–112 (Jan. 1958)

In a rapid and sensitive method for determining small amounts of pyrophosphate (< 1%) in presence of orthophosphates, the complexing effect of pyrophosphate on iron is measured by the 1:10-phenanthroline colorimetric method for iron. L.T.W.

Separation of Non-ionic Surface-active Agents from Mixtures with Anionics by Batch Ion Exchange
M. J. Rosen
Anal. Chem., **29**, 1675–1676 (Nov. 1957)

Non-ionic surface-active agents can be separated from mixtures with anionics by a batch ion-exchange method using a strong anion-exchange resin. Anionic material is

adsorbed by the resin; non-ionic material is washed out with water and methyl alcohol and recovered in substantially quant. yield. L.T.W.

Titrimetric Determination of Sulphamic Acid
C. L. Whitman

Anal. Chem., **29**, 1684–1685 (Nov. 1957)

A method has been developed for the titrimetric determination of small amounts of sulphamic acid or nitrous acid in presence of nitric acid. Sulphamic acid is treated with an excess of sodium nitrite; the excess nitrite is then oxidised with ceric ammonium sulphate, and the excess ceric ion titrated with ferrous ammonium sulphate. From the relationship between the amount of ferrous ion required for titration of the sample and that required for titration of two different blanks, it can be determined whether nitrous or sulphamic acid is present, and the amount of either can be calculated. L.T.W.

Spectrophotometric Determination of Nitrite and Thiourea
K. Hutchinson and D. F. Boltz

Anal. Chem., **30**, 54–56 (Jan. 1958)

The reaction between nitrous acid and thiourea to form thiocyanate is the basis of a simple and rapid colorimetric method for the determination of either nitrite or thiourea. By measurement of the absorbance of the ferric thiocyanate complex, the method can be applied to the determination of 2–12 p.p.m. nitrite and 4–32 p.p.m. thiourea. L.T.W.

Fluorimetric Determination of 1- and 2-Naphthol in Mixtures
D. M. Hercules and L. B. Rogers

Anal. Chem., **30**, 96–99 (Jan. 1958)

Determination of 1- and 2-naphthol in mixtures may be carried out by submitting the mixture in 0·1 M-NaOH to an exciting radiation of 365 mμ. and measuring the emittance at 480 and 426 mμ. for 1- and 2-naphthol respectively. Naphthalene, 2-chloronaphthalene, and 2-naphthalenesulphonic acid do not interfere in thirtyfold excess. No significant errors are caused by the presence of oxygen in the solution, small temperature changes, or moderate exposure to ultraviolet radiation. L.T.W.

Titration of Aromatic Nitroamines as Acids
J. S. Fritz, A. J. Moye, and M. J. Richard

Anal. Chem., **29**, 1685–1688 (Nov. 1957)

Aniline substituted in the 2:4 or 2:4:6 positions with at least two nitro groups, or with one nitro group and one or more chloro groups, can be titrated as acids with butyltriethylammonium hydroxide in pyridine as solvent. Diphenylamine derivatives with at least one nitro group in the *para* position can be titrated in a similar manner. Trinitrotoluene and trinitrobenzene can also be titrated as monobasic acids. In several cases a selective determination of the different components in a mixture is possible. L.T.W.

Refractometric Method for Production Control of Intermediates and Dyes. I—Determination of *p*-Nitrochlorobenzene in *o*-Nitrochlorobenzene
M. A. Portnov and L. A. Muzychenko

Zhur. anal. khim., **12**, 406–410 (1957): *Chem. Abs.*, **52**, 2411 (10 Feb. 1958)

The melting diagram, change in sp.gr. with change of composition of the mixture at 30–90°C., *n* isotherms at 40, 50 and 70°C., temperature coefficient of *n* and polytherms of the pure component have been determined for the system *p*- + *o*-nitrochlorobenzene. The temperature coefficient of the sp.gr. was 0·001, that of *n* at 40–90°C. was 0·0005. Change in *n* with change in composition was additive; change in the *p*-nitrochlorobenzene content in *o*-nitrochlorobenzene changed the *n* by 0·000018. A refractometric method of analysis with an accuracy of ± 1% is described. C.O.C.

Analysis of Phthalic Anhydride
E. E. Shkol'man and N. M. Voroshilova

J. Appl. Chem. U.S.S.R., **30**, 1716–1719 (Nov. 1957)

Esterification of phthalic anhydride with freshly prepared absolute alcohol goes to completion. The wt. of the anhydride in a sample can be calculated from the formula—

$$x = \left(\frac{v}{g} - \frac{v_1}{g_1}\right) \times 0{\cdot}07406 \times 100$$

(x = % wt. of phthalic anhydride; v and v_1 = ml. of 0·5 N-NaOH used for titration of g and g_1 grams of anhydride before and after esterification. T.Z.W.

Identification of Azoic Coupling Components by Microsublimation
W. Kunze

Textil Praxis, **12**, 1253–1255 (Dec. 1957)

A microscope with a heated stage is used for examination. Individual coupling components are characterised by temperature of sublimation, appearance of sublimate, and melting point. The paper is illustrated with 24 photomicrographs. L.A.T.

Determination of Dyes in Solution
E. Bene and I. Géczy

Magyar Textiltech., 370–372 (1954): *Hung. Tech. Abstr.*, **7** (3), 17–18 (1955): *Chem. Abs.*, **51**, 15133 (10 Oct. 1957)

Many anionic dyes when titrated with cationic compounds form compounds insoluble in water but soluble in some organic compounds. In practice the amount of acid or direct dyes in solution can be determined by titration with hexadecylpyridinium bromide in chloroform solution. On titration the dye migrates from the water into the chloroform. At the end of the titration one drop (0·015 ml. with a microburet) of the titrating solution suffices to separate sharply the two liquids, the aqueous solution becoming colourless. The experimental error is ≯ 1%. C.O.C.

Recent Advances in the Identification of *leuco*-Anthocyanins and the Chemistry of Condensed Tannins
D. G. Roux

Nature, **180**, 973–975 (9 Nov. 1957)

The use is described of toluene-*p*-sulphonic acid as chromogenic reagent for developing chromatograms of wood extracts. The formation of phlobaphene, which tends to obscure the chromatogram, is avoided, and *leuco*-anthocyanins of low R_f values can readily be detected. Examples are given from the chromatograms of black wattle (from the bark of *Acacia mollissima*) and quebracho (from the heartwood of *Schinopsis balansae*). A.J.

Identification of Pigments by Back Scattering of β-Rays
K. Yamasaki, K. Saito, and F. Yamasaki

Sci. Papers Japan. Antiques Arts & Crafts (13), 34–36 (1956): *I.I.C. Abstr.*, **1**, Abstr. No. 956 (1957): *Chem. Abs.*, **52**, 1855 (10 Feb. 1958)

The back scattering values are China clay (C.I. Pigment White 19) 118, $CaCO_3$ (C.I. Pigment White 18) 106, White Lead (C.I. Pigment White 1) 372, Yellow Ochre (C.I. Pigment Yellow 43) 99, Litharge (C.I. Pigment Yellow 46) 394, Orpiment (C.I. Pigment Yellow 39) 205, Iron Oxide Red (C.I. Pigment Red 101) 120, Cinnabar (C.I. Pigment Red 106) 387, Red Lead (C.I. Pigment Red 105) 403, Azurite (C.I. Pigment Blue 30) 158, and Malachite (C.I. Pigment Blue 30) 167 counts/min. This method can be used for identifying these pigments. C.O.C.

Mode of Action of Fluorescent Brightening Agents and Measurement of their Relative Efficiency
E. Allen

J. Opt. Soc. Amer., **47**, 933–943 (Oct. 1957)

Fundamental facts on fluorescent brightener emission are reviewed in detail. Two visual effects, a blueing and a lightening, are involved, and the former is much the more important. For any one shade of whitener, both effects are proportional to the *effective fluorescence*, defined as the sum of the tristimulus values of the fluorescent light from the dyeing. The latter may be measured by means of a fluorimeter, providing that a correction factor is applied when two different brighteners are being compared. It is shown to be a reliable indication of whitening power, irrespective of shade. This arises from a compensation of the relative visual effects produced by red-shade and green-shade whiteners. R.B.B.

Spectrographic Analysis of Lead Pigments, Lead Oxides, and Metallic Lead
E. J. Dunn and J. P. Cestaro

Off. Dig. Fed. Paint Varn. Prod. Cl., **30**, 183–188 (Feb. 1958)

Weighed samples, dil. 1 : 1 with pure carbon dust, are burned to completion using a hot-tip thin-walled positive

carbon electrode; there should be some residual burning of the electrodes. The method involves the cross-comparison of the analysis of a large number of elements in metallic lead and lead oxide, and these analyses are compared with the pigments produced. It is quickly shown whether these metallic ingredients exist in the metallic lead, are picked up in processing to litharge, or come about during the manufacture of the pigment. Unwanted ingredients added to paints in field-use are also revealed. The spectrographic analysis of pig lead is quite specific and often characterises the brand of lead.
J.W.D.

Hiding Power and Colour Measurement
D. H. Parker

Paint Ind. Mag., **72**, (10), 18, 20, 22, 24, 26, 28, 30, 47 (1957): *Chem. Abs.*, **52**, 1645 (25 Jan. 1958)

Discussion of methods of determining the hiding power of paints and means for measuring, matching and specifying colour. Included are: reflection and refraction, mechanism of hiding, methods of measuring hiding, colour of solids and liquids, colour standards and matching, colorimetry and systematic collections of colour standards.
C.O.C.

Mathematical Analysis of the Precision in determining Hiding Power
P. Mitton

Off. Dig. Fed. Paint Varn. Prod. Cl., **30**, 156–182 (Feb. 1958)

Typical standard deviations (S.D.) associated with measurements of film thickness and reflectance of paint films on hiding-power (H.P.) charts are discussed, and their effect on the precision of H.P. measurements (in sq. ft. per gal. (U.S.A.) at a 0·980 contrast ratio) is computed. This mathematical analysis is made by differentiating the Kubelka–Munk equations for H.P. (*Zeitschr. Tech. Physik*, **12**, 593 (1931); *J. Opt. Soc. Amer.*, **38**, 448 (1948)) as modified by Switzer (*ASTM Bull.*, No. 197, 60 (1954); *ibid.*, No. 181, **75** (1952)). For given S.D. in measuring film thickness and reflectance, the precision of determining H.P. is poorer when the paint is of low reflectivity and when it has a high scattering coefficient. The effect of the S.D. in film thickness is larger when the paint is applied in a thin film to the chart; the effect for reflectance is larger when the paint is applied in a thick film. If the reflectance of the white substrate itself is measured with the same precision as the reflectance of the paint, the effect of its S.D. may generally be neglected. General recommendations are made regarding optimal conditions for measuring H.P. The equations given allow specific recommendations to be calc. to fit the specific requirements of individual laboratories. It is also shown how to compute the precision with which the original measurements must be made to secure a specified precision in H.P.
J.W.D.

Simple Pigment-settling Gauge and a Simple Anti-sag Test
T. C. Patton

Off. Dig. Fed. Paint Varn. Prod. Cl., **29**, 10–25 (1957): *Chem. Abs.*, **51**, 17195 (20 Nov. 1957)

A perforated disc, used as a probe, is pushed down through the paint by loading with weights with increments at definite time intervals. The vertical height through which the probe moves is taken as an arbitrary measure of pigment settling. An anti-sag test consists of a drawn-down film of paint on a Morest chart. By following the carefully outlined arbitrary procedure in both tests, pertinent information can be obtained. Results are plotted of tests with several antisettling and anti-sag ingredients in various types of paints.
C.O.C.

Electron Microscope Study of Accelerated and Natural Weathering
S. B. Twiss, W. L. Weeks, and D. M. Teague

Off. Dig. Fed. Paint Varn. Prod. Cl., **30**, 7–36 (Jan. 1958)

Electron micrography has been used to study the surface changes produced by the weathering of automobile finishes. A chalk-prone, inorganic-pigmented finish, and a chalk-prone organic-pigmented finish, were both investigated in both lacquers and enamels under Florida exposure and in the XW Weather-Ometer. The findings are discussed, with the aid of 36 electron micrographs, in detail. Natural and accelerated weathering produce similar results, but the latter is less severe, particularly with respect to washing and erosive effects.
J.W.D.

New Preparatory Methods in Electron Microscopy and their Application in Fibre Research
P. Kassenbeck

Melliand Textilber., **39**, 55–61 (Jan. 1958)

Methods of preparation of replicas of fibre surfaces and fibre cross-sections are described. Examples are given of their application in the study of the fine structure of spherulites in polyamide fibres and the skin and core of viscose rayon. Electron micrographs are included.
L.A.T.

Detection of Oiticica Oil in Coatings
G. G. Esposito and M. H. Swann

Anal. Chem., **29**, 1861–1862 (Dec. 1957)

Oiticica oil can be detected in coating materials by the red colour developed with *m*-dinitrobenzene. The reagent reacts with the characteristic carbonyl group of licanic acid, the principal acid constituent of oiticica oil.
L.T.W.

Tracer Techniques in Fibre Research
H. J. White

Anal. Chem., **29**, 1744–1747 (Dec. 1957)

A brief review of the applications of tracers in fibre research is given. Although emphasis is primarily on textile applications, some examples involve paper and biological systems.
L.T.W.

Compressional and Absorptive Behaviour of Bulk Fibre Systems
I. M. Gottlieb, H. Wakeham, and H. M. Virgin

Text. Research J., **28**, 41–46 (Jan. 1958)

Methods are presented for the determination of (*a*) the interfibre volume of fibres in bulk form when immersed under pressure in water, and (*b*) the volume of water which can be absorbed by systems of initially dry fibres at various pressures. Data are given for many fibres.
S.B.D.

Sorption of Moisture by Polymers. I—Effect of Crystallinity
L. Valentine

J. Polymer Sci., **27**, 313–333 (Jan. 1958)

It is concluded that, if a dry polymer can form well defined crystallites, these crystallites will usually be inaccessible to water vapour. Methods of estimating the sorption of water vapour by the amorphous form of a polymer are discussed. In one method the polymer is prepared in varying degrees of crystallinity, and the relationship between sorption and crystallinity extrapolated to zero crystallinity. A variant of this method involves measurement of the accessibility to some simple reagent, e.g. D_2O, which reacts only in non-crystalline regions, the relationship between sorption and accessibility being extrapolated to 100% accessibility. A second main method is based on the fact that conversion of a partially crystalline polymer into a random copolymer frequently destroys crystallinity over a wide range of composition within which there is a linear relationship between sorption and composition. Extrapolation enables sorption of the completely amorphous polymer to be found. This method is applied to several polymers, including cellulose and wool.
W.R.M.

Mercerising Technique to establish "True" Length of Cotton Yarn
C. F. Goldthwait and A. L. Murphy

Text. Research J., **28**, 15–21 (Jan. 1958)

A procedure is suggested for the determination of the length at which a yarn in a skein can be regarded as straightened but not stretched. This requires the determination of the tensions developed in a series of mercerisings of skeins which are alike but are set in the mercerising apparatus at lengths which differ by small percentages. When the maximum tensions attained are suitably plotted, a change in slope in the curve indicates a point which corresponds with the desired length. The change of slope appears to pinpoint a fundamental change in the response of cotton yarn under increasing tension just as it begins to stretch. It is also suggested that a measuring tension for single threads derived from results with skeins may be suitable for determining the initial lengths of yarns for some of the common physical tests such as those for elongation and twist.
S.B.D.

Estimation of the Glass Temperature of Polyamides
G. Bodor
Faserforsch. und Textiltech., **8**, 470–471 (Nov. 1957)
An apparatus is described for determining the specific volume of fibres as a function of temperature. A break in the graph of specific volume against temperature gives the glass temperature. W.R.M.

Testing the Setting of Polyamide Fibres by Fluorescence Microscopy
W. Bobeth and L. Kändler
Faserforsch. und Textiltech., **8**, 444–447 (Nov. 1957)
The setting condition of normally drawn polyamide fibres dyed with Rhodamine G (C.I. Basic Red 8) is estimated by examination with a fluorescence microscope. The method distinguished samples set by hot air and steam. W.R.M.

Qualitative Tests for the Identification of Polyamide and Polyester Fibres with special reference to Rilsan (11-Nylon)
M. Stratmann
Z. ges. Textilind., **59**, 981–982 (5 Dec. 1957)
Use is made of the solubility of fibres in different reagents. Thus, Rilsan is insoluble in cold 6 N. sulphuric acid but is soluble in boiling *o*-dichlorobenzene. With 6- and 6,6-nylons the converse is true. L.A.T.

Redox Titration Curves of Vat Dyes
S. Hafenrichter
Melliand Textilber., **38**, 1257–1263 (Nov. 1957)
Measurement and practical significance of redox potentials of vat dyes are described. The redox titration curves show how the dye uptake is affected by modification of the caustic soda concentration, dyeing temperature, and electrolyte concentration. They also show the effect of using different liquor ratios and dye concentrations. L.A.T.

Testing Rabbit Hair, Wool, and Coarse Animal Hairs for Felting and Milling Properties, Dyeing Behaviour, and Quality of Felt
H. G. Fröhlich
Textil Praxis, **13**, 72–76 (Jan. 1958)
Experimental details and discussion of the results of the following test methods are given—solubility in alkali (0·025 N-NaOH), solubility in acid of increasing concn. (from 0·1 to 2·4 N-HCl), pyridine number (0·5% aq. pyridine by vol.), and potassium permanganate number (0·1 N-$KMnO_4$). L.A.T.

Dye-uptake Meter for Studies of Dyeing Kinetics
E. Bakker and D. Patterson
J.S.D.C., **74**, 168–172 (March 1958)
An apparatus for measuring the rate at which dye is transferred from dyebath to fibre is described. It differs from previous forms of apparatus in that no special device for circulating the solution through a separate observation cell is required. Instead, the observation cell is formed within the dyebath by two inverted "periscopes" facing each other at a fixed distance. A simple double-beam optical system provides a means of eliminating the effects of any drift in the output of the light source or in the sensitivity of the light-measuring system. The types of experiment which can be carried out with the apparatus are mentioned, and some examples given. AUTHORS

Objective Colour Measurement
W. Binder
Melliand Textilber., **38**, 1398–1405 (Dec. 1957); **39**, 77–80 (Jan.); 187–190 (Feb. 1958)
The problem of colour measurement is discussed from the viewpoint of the dyeing technologist. An accurate physical and mathematical basis for such measurement is provided by the German Standard DIN 5033. A general survey of this field is given and the possiblities, and the limitations of colour measurement are defined. L.A.T.

Colour-naming Experiments for Red and Green Monochromatic Lights
M. A. Bouman and P. L. Walraven
J. Opt. Soc. Amer., **47**, 834–839 (Sept. 1957)
Colour appreciation for normal trichromats is studied at very low illumination levels, such as may occur in the perception of coloured signal lights. Colour naming is erratic under these conditions, and this is shown to be related to fluctuations in the number and distribution of light quanta reaching the retina. R.B.B.

Fluorescence of the Crystalline Lens and Electroretinographic Sensitivity Determinations
E. Dodt and J. B. Walther
Nature, **181**, 286–287 (25 Jan. 1958)
Fluorescence appears to account for the difference between electroretinogram and sensory spectral sensitivity curves. Electroretinogram sensitivity data for normal and lensless eyes are given. R.B.B.

Correlate for Lightness in Terms of C.I.E. Tristimulus Values—II
G. Wyszecki and C. L. Sanders
J. Opt. Soc. Amer., **47**, 840–842 (Sept. 1957)
The second-degree equation relating lightness index to C.I.E. X, Y, Z values, derived previously (idem, *ibid.*, **47**, 398 (1957): J.S.D.C., **74**, 113 (Feb. 1958)) is tested and shown to be satisfactory. The influence of background on lightness index is studied. R.B.B.

Chromatic Flicker Fusion Frequency as a Function of Chromaticity Difference
C. V. Truss
J. Opt. Soc. Amer., **47**, 1130–1134 (Dec. 1957)

Colours produced by Reflection at Grazing Incidence from Rough Surfaces
W. E. K. Middleton and G. Wyszecki
J. Opt. Soc. Amer., **47**, 1020–1023 (Nov. 1957)

Colour Correction of Paint by Tristimulus Colorimetry
Los Angeles Paint & Varnish Production Club
Off. Dig. Fed. Paint Varn. Prod. Cl., **28**, 624–632 (1956): *Chem. Abs.*, **51**, 18638 (25 Nov. 1957)
A sample of paint is tested in a spectrophotometer for absorption and scatter coefficients. Three readings are taken with green, amber, and blue Hunter filters respectively. The total reflectivity is determined by the Kubelka–Munk equation $(A/S) = (1 - R)^2/2R$ (Judd, *Color in Business, Science and Industry* (1952) 316). Values for R can be obtained from published tables. These values are correlated with the concentration of individual pigments in the equation—

$$(A/S)_{\mathrm{mixt}} = [C_1A_1 + C_2A_2...]/[C_1S_1 + C_2S_2...]$$

(C = conc. of the individual pigment). Three simultaneous equations are obtained, one for each filter. These are solved by a specially designed mechanical slide rule. Amounts of each pigment to be added to the bulk of the paint to obtain correct colour matching are thus determined. The constants for each pigment have been determined previously. Results with yellow and red iron oxides (C.I. Pigments Yellow 42 and 43 and Red 101) and TiO_2 (C.I. Pigment White 6) agree with the theory, those with Chrome Yellow (C.I. Pigment Yellow 34) and Phthalocyanine Blue (C.I. Pigment Blue 15) do not agree. C.O.C.

Space Error in Colour Matching
R. W. Burnham, J. R. Clark, and S. M. Newhall
J. Opt. Soc. Amer., **47**, 959–966 (Oct. 1957)
No significant differences were observed between matches with a tristimulus colorimeter, in which test and matching stimuli could be made to occupy different relative positions (left or right, top or bottom) in a symmetrical 2° bipartite field, with a surrounding 71° white field. R.B.B.

Comparative Light-fastness Tests
L. F. C. Friele and H. J. Selling
Mededeling van het Vezelinstituut, No. 115 (1957): *Melliand Textilber.*, **38**, 1269–1273 (Nov. 1957)
Results are described of comparative assessments of fastness to light of dyed patterns after exposure in daylight and in the Xenotest, Fugitometer, C.P.A. fading lamp and Fade-Ometer. The results obtained with the Xenotest agree closely with daylight exposures, and the latest instruments are suitable for standard light fastness testing. The temperature of the test pattern should not exceed 50°C., and an effective humidity of 30% is recommended. L.A.T.

Study of Dyes and Fibres during Fading— Chemical Transformation of *p*-Aminoazobenzene by Ultraviolet Radiation
I. Rusznák and M. Fehérvári
Magyar Textiltech., 127–129 (1954): *Hung. Tech. Abstr.*, **7** (1), 25–26 (1955): *Chem. Abs.*, **51**, 15134 (10 Oct. 1957)

Dyeings and solutions of Chloramine Sky Blue FE and of 5 similar dyes were exposed to ultraviolet radiation which caused both dyeings and solutions to fade. Examination showed that the dyes are decomposed into both substantive and non-substantive components. One portion of *p*-aminoazobenzene was oxidised with $K_2Cr_2O_7$ and another exposed to ultraviolet radiation and examination showed the same decomposition products. These results indicate that irradiation affects dyes in the same way as oxidation. If cotton and rayon are irradiated before dyeing then the dyeings are not as fast to light as those on non-exposed fibres; dyeings on wool, on the contrary, are faster to light if the fibres have been irradiated before dyeing. C.O.C.

Instrument for selecting visually Neutral Photographic Dye Images
A. J. Sant and O. E. Miller
J. Opt. Soc. Amer., **48**, 4–8 (Jan. 1958)

The design and the construction are described of an instrument enabling an observer to select a colour-film neutral with high precision. R.B.B.

Band Systems for Appraisal of Colour Rendering
B. T. Barnes
J. Opt. Soc. Amer., **47**, 1124–1129 (Dec. 1957)

The use and the limitation of the "band system" method for comparing the colour-rendering properties of fluorescent light sources is discussed. R.B.B.

Colour Chart for Evaluating Quality of Colour Reproduction
Phot. Sci. & Eng., **1**, 74–78 (1957): *Chem. Abs.*, **52**, 2624 (25 Feb. 1958)

A description of materials and construction of a chart used to facilitate quantitative evaluation of the quality of colour reproduction obtained by various photographic processes. It includes 9 saturated, 7 achromatic, and 8 desaturated colours. Permanent pigments are used to enable the chart to be used in direct sunlight without fading or discolouring. C.O.C.

Electroanalysis with Controlled Cathode Potential of Metallic Copper applied to Fabrics as Metallo-organic Fungicides
J. Bubernak and A. D. Baskin
Text. Research J., **27**, 878–882 (Nov. 1957)

A method is described for the determination of Cu in fabrics treated with organo-copper fungicides. The samples investigated contained fire retardants as metallic oxides, among which were those of Sb, Ca, Zn, and Fe. Antimony offers the greatest interference in the electrolytic determination of copper, but this interference was eliminated by depositing the Cu from ammonium tartrate soln. with careful control of the cathode potential, using a laboratory potentiometer to measure the voltage. Data are presented showing that Cu may be electroplated from ammonium tartrate soln., containing a sixfold greater concn. of Sb, with a cathode potential of −0·6 v. Results are also given for analyses of fabrics containing copper 8-quinolinolate and copper naphthenate singly or in mixture. A rapid and safe method for digesting cloth samples with sulphuric and perchloric acids is described. S.B.D.

Determination of Silicon in Textile Materials
G. D. Nessonova, D. V. Turkovskaya, and É. D. Mosolova
Tekstil. prom., **18**, 55 (Jan. 1958)

The silicon content of textiles which have undergone finishing with silicon-organic compounds is checked satisfactorily by chemical analysis. A recipe for the method of wet oxidation (conc. HNO_3, then conc. H_2SO_4) is given, the complete analysis requiring 2 hr. G.J.K.

Determination of α-Epoxides containing a Tertiary Carbon Atom via Catalytic Isomerisation with Zinc Bromide
A. J. Durbetaki
Anal. Chem., **29**, 1666–1668 (Nov. 1957)

A method has been devised which makes possible the determination of α-epoxides containing a tertiary carbon atom which cannot be determined by any method hitherto described. The α-epoxide is converted into its isomeric aldehyde by catalytic isomerisation with zinc bromide in benzene; the aldehyde is determined gravimetrically with 2:4-dinitrophenylhydrazine. L.T.W.

Detection of Urea, Melamine, *iso*Cyanate, and Urethane Resins. Rapid Group Test for Nitrogen, Silicon, Phosphorus, and Titanium
M. H. Swann and G. G. Esposito
Anal. Chem., **30**, 107–109 (Jan. 1958)

In specific tests for urea–formaldehyde and melamine–formaldehyde coating resins, the sample is refluxed with a mixture of acetic acid and acetic anhydride containing *p*-dimethylaminobenzaldehyde. In presence of urea resins a blue colour is formed, while melamine resins form an insoluble condensate. Nitrogen, phosphorus, silicon, and titanium are detected rapidly in coating materials by a new microprocedure. L.T.W.

Simplified Control Analyses of Solutions used in Partial Acetylation of Cotton
E. M. Buras, A. S. Cooper, and M. D. Cruz
Anal. Chem., **30**, 104–107 (Jan. 1958)

Volumetric methods have been developed for the rapid control analysis of mixtures of acetic acid and perchloric acid with acetic anhydride and with water which occur in the three-component baths used in the partial acetylation of cotton. Perchloric acid is titrated in glacial acetic acid with potassium hydrogen phthalate using *p*-naphtholbenzein as indicator. Water and acetic anhydride are determined empirically by phase separation in the presence of a large volume of hydrocarbon solvent. L.T.W.

Titration of Hydrogen Sulphide and Sulphides with Organic Mercury Compounds
M. Wroński and B. Philipp
Faserforsch. und Textiltech., **9**, 36–37 (Jan. 1958)

Organic mercury compounds of the type $C_6H_5{\cdot}Hg{\cdot}X$, where X is OH or Cl, react quantitatively with sulphur compounds containing the SH group. Titration with a solution of $o\text{-}C_6H_4(COOH){\cdot}HgOH$ is used to estimate dissolved sulphur in liquors met in the cellulose and viscose industries. The reagent can also be used to estimate hydrogen sulphide in air and waste gases. W.R.M.

Determination of Xanthate Sulphur in Viscose
J. P. Dux and L. H. Phifer
Anal. Chem., **29**, 1842–1845 (Dec. 1957)

In a new and rapid method for determination of cellulose xanthate in viscose, undiluted viscose is forced through a specially constructed anion-exchange column to remove other sulphur-containing compounds. The sample is then weighed and diluted, and the cellulose xanthate determined by measuring the ultraviolet absorbance at 303 mμ. L.T.W.

Estimation of Acidity in Cellulose— Reaction of Crystal Violet Base (C.I. 42555B) with Cotton
M. Rebek and H. Baumgartner
Mh. Chem., **88**, 956–966 (Dec. 1957)

The estimation of acidity in cellulose is critically discussed. Concentration-dependence and time-dependence of the reaction of cellulose with Crystal Violet base in benzene are studied, and some results obtained with standardised cotton samples are given. W.R.M.

Colour Variation in Paperboard
J. T. Patterson, S. L. Goldheim, and W. M. Hancock
Tappi, **41**, 20–23 (Jan. 1958)

By means of a Colormaster differential colorimeter, a control chart system has been devised to help mills specify and control the colour variations in white paperboard. The necessary data are taken directly from the Colormaster readings without resorting to extensive calculations or absolute values. Extent and direction of colour variation are readily apparent from the control chart. R.A.

Determination of Sulphate Acid Ester in Unstabilised Cellulose Nitrate by the Hydrolysis Method
P. E. Gagnon, K. F. Keirstead, and B. T. Newbold
Canadian J. Chem., **36**, 291–295 (Feb. 1958)

The precision of duplicate determinations of sulphate acid ester by hydrolysis in acetone–water solution is evaluated. The sulphate acid ester content of both unstabilised wood and cotton cellulose nitrates increases with decreasing nitrogen content. W.R.M.

Determination of Carbonyl Groups in Modified Celluloses—Comparison of the Borohydride and Copper Number Methods
S. Lidman-Safwat and O. Theander
Svensk Papperstidning, **61**, 42–46 (31 Jan. 1958)
A linear relation exists between copper number and carbonyl content, but the ratio differs for different types of celluloses. The structure of the reducing unit affects the copper uptake. For greater accuracy with low carbonyl contents a modified borohydride method was used with larger vessels, more dilute reagent, longer reaction times, and pH 9·6–9·8. The method is rapid, non-stoichiometric, and gives reproducible results under strictly standardised conditions. R.A.

Determination of Fixed Chrome and Iron in Leather (with Chrome- and Iron-containing Pigment Finishes)
A. Simoncini and M. Gelsomino
Cuoio, Pelli, Materie concianti, **33**, 7 (1957): *J. Soc. Leather Trades Chem.*, **42**, 64 (Feb. 1958)
Chemical methods of dissolving the tanning agent without dissolving the pigment are difficult, whilst buffing off the finish removes an appreciable amount of grain, which may introduce an error in the analysis. Also buffing machines will not cope with the small size of sample normally available. Using the blasting technique with acid-washed white sand in place of carborundum, the pigment finish can be removed from small pieces with a negligible amount of grain. Analyses for chrome and iron before and after finishing were in excellent agreement, though ash contents were slightly high owing to some occlusion of the sand in the grain. C.J.W.H.

Optical Diffractometer for Facilitation of X-Ray Diffraction Studies of Macromolecular Structures
H. N. Wyckoff, R. S. Bear, R. S. Morgan, and D. Carlstrom
J. Opt. Soc. Amer., **47**, 1061–1069 (Dec. 1957)
Construction details are given of a "diffractometer" employing a photographically produced trial structure mask. An optical diffraction pattern produced by the mask with visible light is then compared with the X-ray diffraction pattern which is being interpreted as the trial structure. Structure determinations by this means are described for polypeptide α-helices and collagen and deoxyribonucleate molecules, as well as studies on larger structural units exhibiting small-angle X-ray diffraction, e.g. collagen fibrils. R.B.B.

Use of Birefringence in Determining Fibre Structure and Investigating Dyeing Faults
K. Hájek
Věda a Výzkum v Průmyslu textil., 1, 23–34 (1956): *Chem. Abs.*, **52**, 5828 (10 April 1958)
The birefringance $B = n'-n''$ of a fibre is a measure of orientation in both the crystalline and amorphous regions. Two methods of determining it are described. (1) The immersion method, in which the fibres are immersed in a series of liquids of known n and observed microscopically in monochromatic polarised light until the n of the fibre and the liquid are the same. This is done when the fibre axis is parallel to the plane of polarised light, and again when it is perpendicular, to determine n' and n''. (2) The compensation method, in which the retardation of one refracted beam behind the other is determined by placing the fibre at 45° between crossed Nicol prisms of a polarising microscope, and compensating the interference colours by means of a quartz disc, whose rotation from the horizontal is measured. The fibre thickness must be known. When Silon fibres were stretched to various extents S, B varied rapidly with S at low values of S, but slowly at higher values. The sensitivity was good only up to $S = 125\%$. When investigating the causes of streakiness in dyed viscose rayon fabrics, determination of B was useful in detecting faults caused in production and processing. The absorption of dye decreased with increase in B. Differences in B were more often caused during fibre production than in their subsequent processing. Results of B determinations were parallel to determinations of wet modulus, i.e. strength : elongation ratio of wet fibres. C.O.C.

Wavelength Discrimination for Point Sources
R. E. Bedford and G. W. Wyszecki
J. Opt. Soc. Amer., **48**, 129–135 (Feb. 1958)
Wavelength discrimination curves measured for three small field sizes, 1° and less, establish a definite maximum at 420 mμ. R.B.B.

Observer Differences in Colour-mixture Functions studied by means of a Pair of Metameric Greys
K. L. Kelly
Bur. Stand. J. Res., **60**, 97–103 (Feb. 1958)
Data obtained by 39 normal observers are used to decide whether any set of colour mixture functions gives predictions of the character of the colour difference between two metameric greys. For a 2° field, the 1931 C.I.E. standard observer functions give better agreement than the Judd 1951 and the Stiles 1955 functions. The 1955 Stiles 10° functions agree fairly well. Reciprocal colour-temperatures required for red–green matching a highly metameric and a nearly neutral grey are tentatively taken as approximately characterising an observer's vision. Values for a 10° field are regarded as a measure of lens pigmentations and the differences between 2° and 10° fields as a measure macular pigmentation. Neither measure shows significant correlation with eye or hair colour. R.B.B.

PATENT

Measuring the Lustre of Textile Fabrics and Yarns
British Rayon Research Assocn. *BP* 791,477
Apparatus providing a sensitive and convenient method of carrying out comparative measurements of the lustre of textile fabrics and yarns wound in the form of a pad. C.O.C.

Hydroxydiazoamino Compounds of Anthraquinone (IV p. 493)
Activation Analysis of Fibrous Materials (VI p. 496)
Reflectance Measurements on Dyed and Undyed Viscose Rayon Fabric in the Ultraviolet and Visible Regions (VIII p. 498)
Damage caused in Cellulose by Treatment with the Neutral System Sodium Formaldehyde-sulphoxylate–Hydrogen Peroxide (IX p. 499)

XV—MISCELLANEOUS

Cobaltous Oxide–Titanium Dioxide System and Ceramic Colouring Stains
J. H. Strimple
Univ. Microfilms (Ann Arbor, Mich.), *Publn.* No. 22555, 140 pp.; *Dissertation Abstr.*, **17**, 2541 (1957): *Chem. Abs.*, **52**, 3290 (25 Feb. 1958)

PATENTS

Colouring Glass
Corning Glass Works *USP* 2,779,688
A glass article containing $> 93\%$ SiO_2 is leached in dilute acid. Parts of its surface are then treated with a strong solution of an inorganic glass colorant so as to colour only the surface layer, the solution of colorant then being replaced by water until the absorbed colorant moves by capillary action further into the glass. The article is then dried and fired without fusion to close the pores and incorporate the colorant within the glass. C.O.C.

Colouring of Silver Dragées
J. Harradine & Sons *BP* 791,376
The silver dragées are first coated with shellac or other material yielding a transparent waterproof coating, dried and then treated with an aqueous solution of a colouring material containing sugar and gum to facilitate even wetting of the waterproofed surface of the dragées. C.O.C.

Coloured Crayons for Decorating Ceramics
R. Zgraggen *BP* 793,359
Crayons for use on glazed ceramics before firing consist of a fired mixture of a metal compound, an inorganic carrier and a binder. Thus calcined stannic oxide (C.I. Pigment White 15) (100), $CaCO_3$ (C.I. Pigment White 18) and quartz (C.I. Pigment White 27) (30) are treated with a hot saturated solution of potassium chromate (6) and borax (8), wet ground, dried and heated until the metals are oxidised. The product (8), kieselguhr (2) and 10% aq. gum arabic (1) are made into a paste, formed into crayons in a press, left in the press for several days until hard and then heated to 400–1000°c. This yields red crayons which when used on ceramics release the pigment evenly without damaging the surface of the articles treated. C.O.C.

APPOINTMENTS etc

Advertisements relating to APPOINTMENTS VACANT, APPOINTMENTS WANTED, and MISCELLANEOUS ITEMS are invited for insertion on this page. Advertisements of Appointments Wanted are gratis to members, but must not exceed twenty-four words.

All inquiries relating to Advertisements should be addressed to THE GENERAL SECRETARY, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE.

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viii	Mono Pumps Ltd
xliv	Sir James Farmer Norton & Co Ltd
vii	Samuel Pegg & Son Ltd
vi	L J Pointing & Son Ltd
xxvii	James Robinson & Co Ltd
xi	Sandoz Products Ltd
xiii	Sandoz Products Ltd
ix	Saunders Valve Co Ltd
xxxviii	T Saville Whittle Ltd
v	Shell Chemical Co Ltd
iv	Shirley Developments Ltd
xxviii	F Smith & Co (Whitworth) Ltd
xxxviii	Standard Chemical Co
xlii	Staveley Iron & Chemical Co Ltd
vi	Sun-Ray Dyeing Co (Leicester) Ltd
xxxiii	Tennants Textile Colours Ltd
x	W P Thompson & Co
xxv	Vinyl Products Ltd
xxxvii	West Indies Chemical Works Ltd
xl	J B Wilkinson (Chemicals) Ltd
viii	Williams (Hounslow) Ltd
xxiii	Yorkshire Dyeware & Chemical Co Ltd

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